American Perfumer AND AROMATICS

COSMETICS
TOILETRIES
SOAPS
•
FLAVORS
AEROSOLS
•
ESSENTIAL OILS

APRIL 1960

THE MAGAZINE OF TASTE AND SCENT

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Elsholtzia Christata Wild

NOT MUCH NOISE FROM *Norda*

but...

Quietly, we've been going ahead with important improvements, and with expansion of our facilities to serve you better than ever.

For instance, Norda recently spent over a quarter-million dollars on plant modernization, including new machinery and storage tanks, at our Hanover, New Jersey plant.

Constant progress like this explains, in part, the confidence and trust so many place in Norda for the finest flavors, aromatic chemicals, and essential oils.

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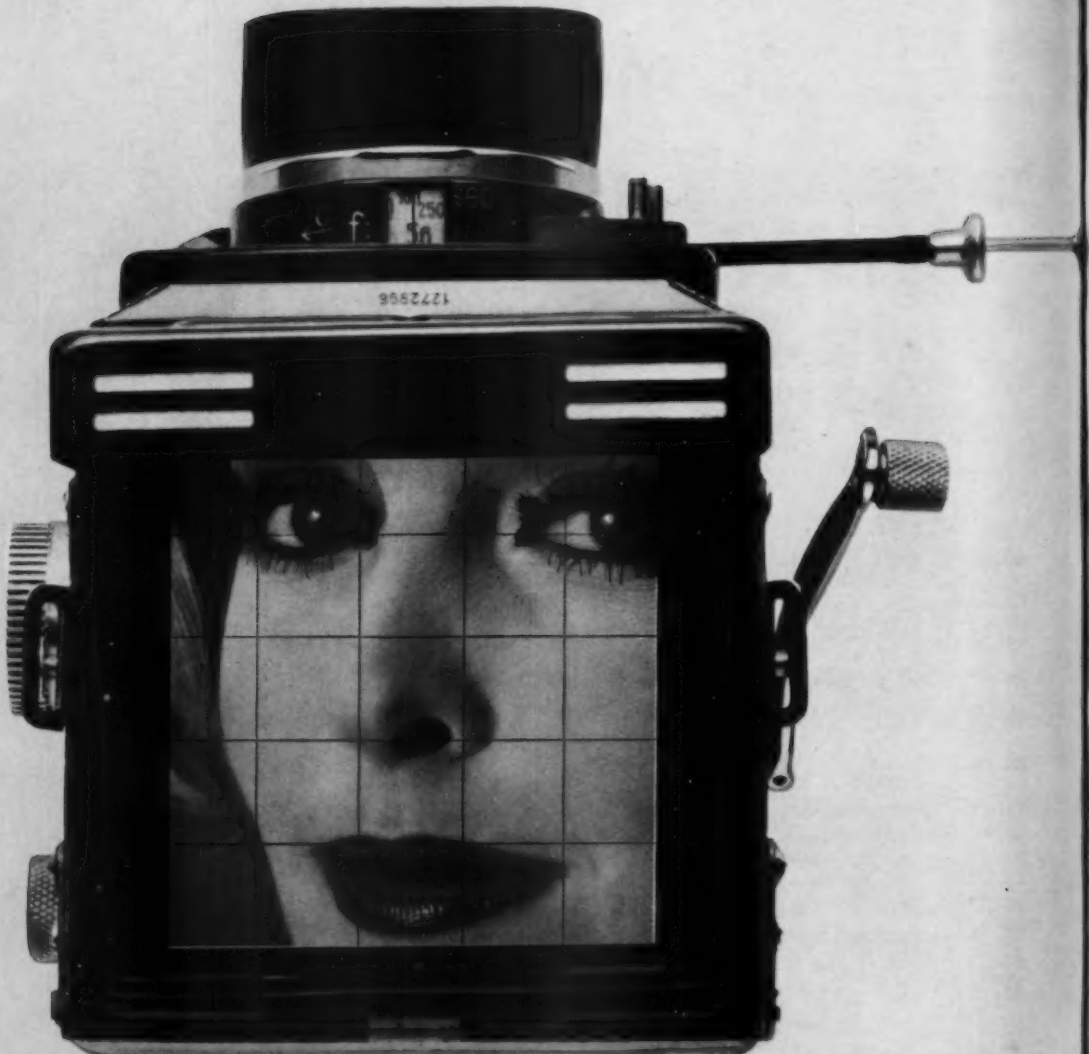

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American Perfumer
AND AROMATICS

VOL. 75, NO. 4 APRIL, 1960

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COVER: A drawing of Labiated Elsholtzia Christata, whose essential oil is just now becoming of commercial importance. This drawing was prepared for the American Perfumer by the staff artist of the Chicago Museum of Natural History. See a story on this oil beginning on page 33.

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Comming in June!

the seventh in a series
of documentaries

PERFUME DOCUMENTARY

Ten original papers have been prepared for this publication, dealing with various phases of perfumery of cosmetic and related products.

The previous documentaries have proved to be prime reference sources in the fields that that covered. In this respect, the Perfume Documentary will take its place as a permanent reference source.

• • •

Also in the June issue, a Salute The Toilet Goods Association, telling of its services to the industry.

• • •

Advertising forms close May 15th. Publication June 15th. Regular advertising rates apply.

Creative Perfumer

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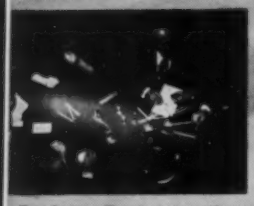
Cosmetic Research



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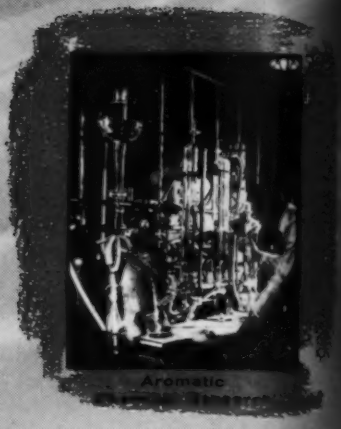

Aromatic

People...

THE IMPORTANT INGREDIENT

Sales

A successful perfume is a combination of the talents of many people . . . each concentrating his specialized abilities to achieve the right fragrance for your product.

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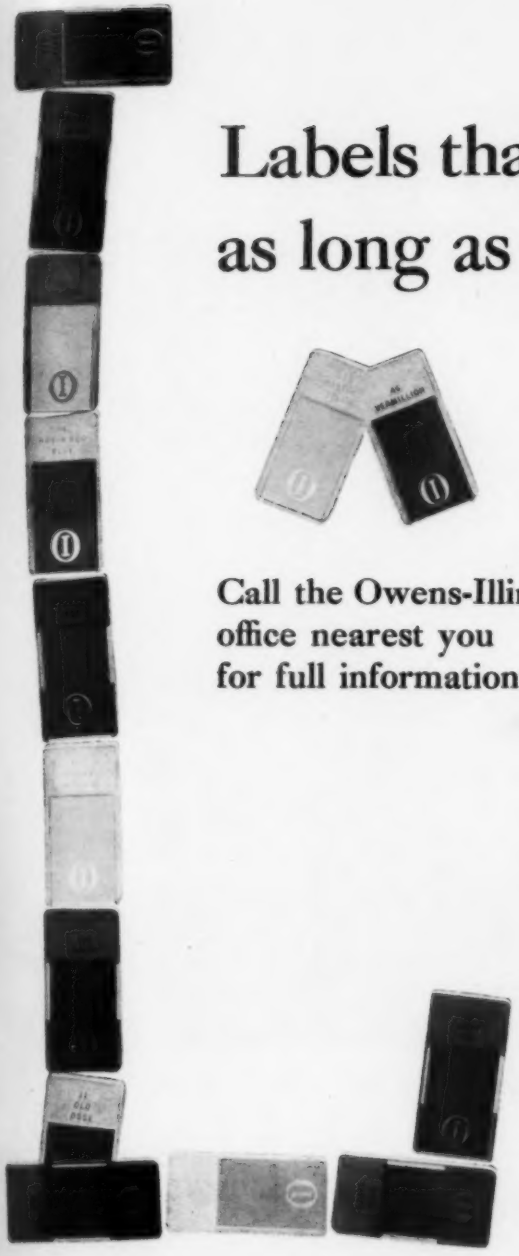

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Particularly interesting variations result when it is combined with the aristocrat of rose essences, Penick's long-lasting Rosottone Supreme.

For colognes, creams, soaps and other cosmetics and toiletries, Penick offers a range of more modestly priced rose florals, including Rosottone* and Rosottone Savon*.

*Penick trademark

Samples of these Penick essences on request.

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Aromatics and Flavors Department

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(LYOPHILISED)

what is it?

Disease free and carefully selected placenta are obtained by aseptic techniques and are quick frozen. Then at the manufacturing site, the blood serum is removed. The residual tissue is subjected to a series of freeze-thaw steps causing the cells to burst, releasing in liquid form all the bio-active stimulating substances, as reported in the literature. Among them are . . .

> Enzymes (primarily alkaline phosphatase, acid phosphatase, oxidase and peroxidase); Amino acids (aspartic acid, glutamic acid, serine, glycocoll, threonine, alaline, tyrosine, valine, lysine, histidine, arginine, oxyproline, phenylalanine, leucine, isoleucine, proline); Mineral ions, especially sodium and potassium; Nucleic acids; Proteins and/or peptones; Also, folic acid, progestin, estrogen, androgen, prolactin, corticosteroids, chorionic gonadotrophins, vitamins, oligo elements, thyrotrophic hormones, pregnanediol-3-a, etc.

This liquid is then dehydrated to a powder by a freeze-dry process. Thereby all of these bio-active components are preserved unaltered. To reconstitute, simply add distilled or de-ionized water.

what will it do?

There is an abundance of scientific literature detailing remarkable effects of this material on normal and pathological skin. Among many others, Michel Prunieras* reports a notable number of remissions in psoriatic conditions. This is only one of many dermatological findings reported. Others deal with senile skins, acne, etc.

Our work at a leading University definitely indicates that most cosmetic preparations or drug bases actually depress skin respiration. Utilizing reconstituted Human Placental Extracts Lyophilised offsets this phenomenon.

write for suggested formulations, etc. . . .

R.I.T.A. CHEMICAL CORPORATION

612 N. MICHIGAN AVE. – CHICAGO 11, ILL. – PHONE SU. 7-0051

* Michel Prunieras, LA THERAPEUTIQUE TISSULAIRE EN DERMATOLOGIE, No. 205, July 1949

LAST MINUTE NEWS . . .

Executive changes at Hazel Bishop

Daniel Van Dyk has been elected chairman of the board of Hazel Bishop Inc. succeeding Raymond Spector who will continue with the company as a major stockholder, a director and a consultant. Herman L. Johnson, formerly executive vice president, was elected president and chief executive succeeding Mr. Van Dyk.

Lewis to head Toni brand promotion

Joseph T. Lewis has been appointed to the newly-created position of director of brand promotion for The Toni Company. Mr. Lewis has been associated with brand activities since he joined the division of The Gillette Company in 1948. He recently has been serving as a brand supervisor of a group of Toni products. In his new position he will supervise advertising, brand promotion and packaging for the company's complete line of products.

Yardley to sponsor telecast of Princess Margaret's wedding

A thirty minute telecast of the wedding of Britain's Princess Margaret and Anthony Armstrong-Jones will be sponsored by Yardley of London, Inc. Carried on ABC-TV the news show will begin at 5 P.M. on May 6th after videotapes are received from Westminster Abbey by jet aircraft. Commercials will be of the Red Roses products which are being featured in the company's print advertising and promotion programs this year.

Mark Twain subject of Breck Showcase TV "Special"

Hal Holbrook, noted for his impersonation of Mark Twain in his one-man show on Broadway last year, will re-create the role on "Roughing It" as the fifth in the 1960 series of Breck Showcase TV "Specials" sponsored by John H. Breck, Inc. The hour-long show will be seen on Friday, May 13th over NBC-TV in color. Products to be advertised on the "Special" will be three shampoos, Hair Set Mist, Creme Rinse, Banish and children's shampoo.

Prince Matchabelli promotes Spray Mist in LIFE magazine

A double-spread, full-color advertisement in the May 2nd issue of LIFE magazine will introduce Prince Matchabelli's new Spray Mist bottle. The container with a plastic coating is partially transparent. Usually the glass bottle has been covered with opaque plastic coating or enclosed in a metal container to disguise the aerosol fill line. This pressure-spray container is a new unique design. A different color bottle is used for each of Matchabelli's Cologne fragrances.

Glass Container Use Up in 1959

The Glass Container Manufacturers Institute has released figures which show an increase of 6.5% in total shipments of glass containers in 1959 from 1958 figures. Categories showing sizable gains were medicinal and health, and toiletries and cosmetics. Medicinal and health supply packers received an increase of 5.3% over 1958 and toiletries and cosmetic bottle shipments rose 7.6% in 1959.

for a safe stick anti-perspirant...

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Completely safe Reheis Chloracel® . . . available in 40% w/w solution or powder . . . is the ideal base for alcoholic stick anti-perspirants. It's non-irritating to the skin . . . won't harm fabrics . . . requires no buffering . . . and is available, in any quantity, for immediate shipment. These are reasons why it's wise to come to Reheis first for a safe *stick* anti-perspirant base.

In fact, it's wise to come to Reheis first for *any* anti-perspirant base. No other supplier produces as many grades. For cream, lotion, spray, or powder formulae, there are *five* grades of Reheis Chlorhydrol® to choose from . . . granular, fine, medium, impalpable, and 50% w/w solution. These, too, are available for immediate shipment in any quantity. For detailed information, write to:

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2947

WAVEMASK 30

works magic with home permanents

The new Synfleur fragrance WAVEMASK 30 can help cure even your most difficult case in home permanent formulation.

WAVEMASK 30, based on essential oils and aromatic chemicals, carefully tested for stability, compatibility and solubility, is an artistic formulation developed out of long and intensive Synfleur research.

In practical application, it has proven itself as practically a panacea to cold wave preparation odor problems. Additionally, it can be adapted as a basic fragrance for effective use in other thioglycolate products requiring similar masking treatment.

The Synfleur laboratories staff will be glad to assist you on your masking problems with samples, data or other help, without obligation.

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Questions and Answers

Q. *I am having trouble with the formulation of cold wave neutralizers. First, when sodium perborate is packaged in laminated foil there is, so we are told, an additive which when added stops the bleaching action of the perborate. What is this chemical? Is it tartaric or citric acid and is this acid compatible when backed with perborate? Secondly, some manufacturers advocate sodium bromate with another power added for packaging, such as an ammonium quartenary. I know something about surfactants but not enough to discern one from another. Is there a common sense book about these things for the ordinary, untrained chemist? B. F. S., N. Y.*

A. The only thing with which we are acquainted that can stop the bleaching action of any "per" compound is an enzyme called "catalase." This is usually sold in a solution and is applied to the hair after the oxidation has been allowed to carry on as far as is wanted. The application of catalase stops the oxidation immediately at that point. To our knowledge, it is not possible to pack the catalase with the perborate. As for the use of sodium bromate, this material has been in use for a great many years. We have not seen a package containing a quarternary compound such as you mention but see no reason why one could not be used. We suggest that you approach the manufacturers of such quarternaries: Onyx Oil and Chemical Co., 15 Exchange Place, Jersey City 2, N. J.; Stepan Chemical Co., 427 West Randolph St., Chicago 6, Ill.; Rohm & Haas Co., Washington Square, Philadelphia, Pa. We are afraid there is not much of a common sense book on surfactants such as you want. If you had been a subscriber to the AMERICAN PERFUMER during the past 12 months, you would have received a copy of the documentary on commercial surfactants which covered practically each type that is commercially offered. A good deal of the data given in this was in common sense form.

Q. *Please advise by literary recommendations (the more succinct and understandable to the layman, the better) as to the best approaches to the following problems and their solutions created by allergenic products in drug and cosmetic manufacturing: 1. allergy testing; 2. allergy law suits; 3. allergy insurance—if any. N. K., Ohio.*

A. The allergenisity of materials or finished products is not usually determined by a layman. This is done by the pharmacologist especially trained to do this type of work. Generally, the work is first done on animals and is then supplemented, if needs be, on man himself. There are a number of laboratories that do this sort of work, and we are pleased to give you the names of the Laberco Laboratories, 123 Hawthorne St., Roselle Park, N. J., and Rosner-Nixson Laboratories, 7737 South Chicago Ave., Chicago 19, Ill. If your public library has it, you can find the number of people who have instituted law suits resulting from allergic responses to a manufacturer's product in the Prentice-Hall Reports on product liability cases. As to insurance on allergy: of course, you cannot get insurance against allergies since there is no known non-allergic material on this earth. However, you can get product liability insurance and this you can request from any company selling liability insurance in your area. If they themselves do not handle it, they no doubt can suggest someone who does.

Q. *We intend to start the manufacture of powder and cream for artificial teeth as well as cleansing agents for these. Would you please advise where to find literature and descriptions of such preparations? J. M., Sweden*

A. Sagarin's book, *Cosmetics: Science and Technology* would be the best one we could recommend to you for data on such preparations. Basically, a dentifrice for artificial teeth need not be any different than that for one's own teeth. In general, however, the powders used are somewhat more alkaline, containing such ingredients as the polyphosphates which act as cleaners as well as "sterilizers."

From time to time suggestions have been and will be made in this magazine with respect to processes, devices, materials, appliances, equipment and the like. It is not practicable for the writers and editors to have a patent search or examination made in connection with each such suggestion. Our readers are, therefore, requested and indeed urged to determine for themselves whether any patent or other right will be violated before acting on any such suggestions.

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VERONOL
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An Acetal of fine character, great lift and extreme stability. Ideal for all rose and floral compounds for soaps and cosmetics.

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Green note, desirable in many compositions for top notes. Stable in soaps and economical.

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A special natural rose base processed by extraction from flowers other than Rose de Mai.

CHENIRAX
An extraction of Mousse de Chene and Gum Styrax.

IRIS ED 205
An interesting and economical Iris body distilled over Iris Roots.

DISTIRONE
A reconstitution of Absolute Iris (Irone) distilled over Iris Roots.

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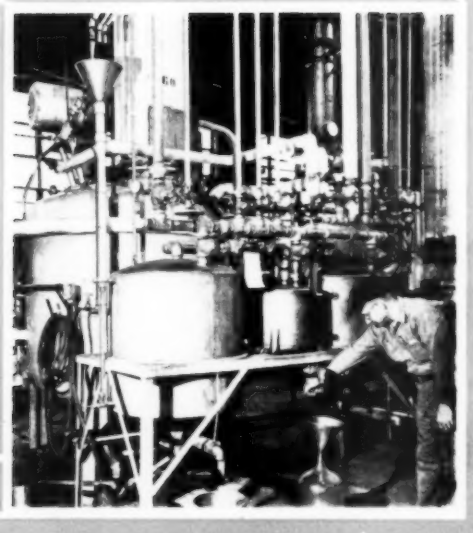

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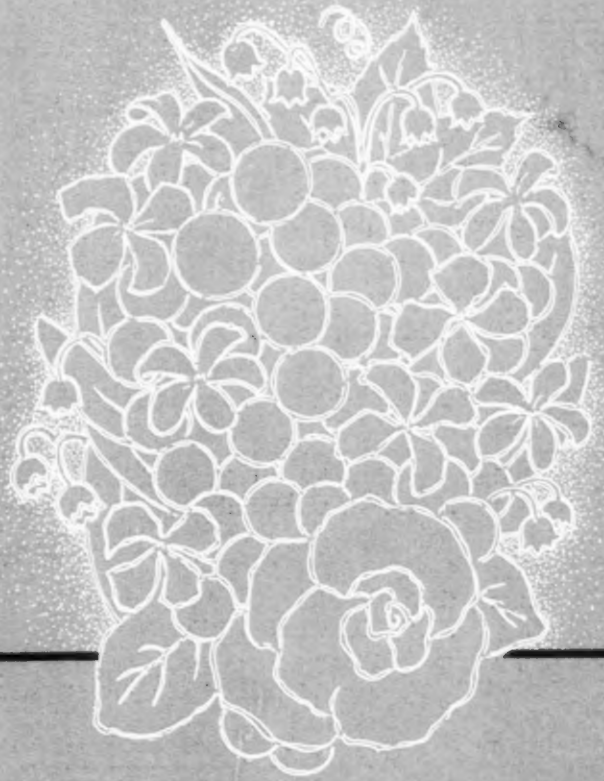

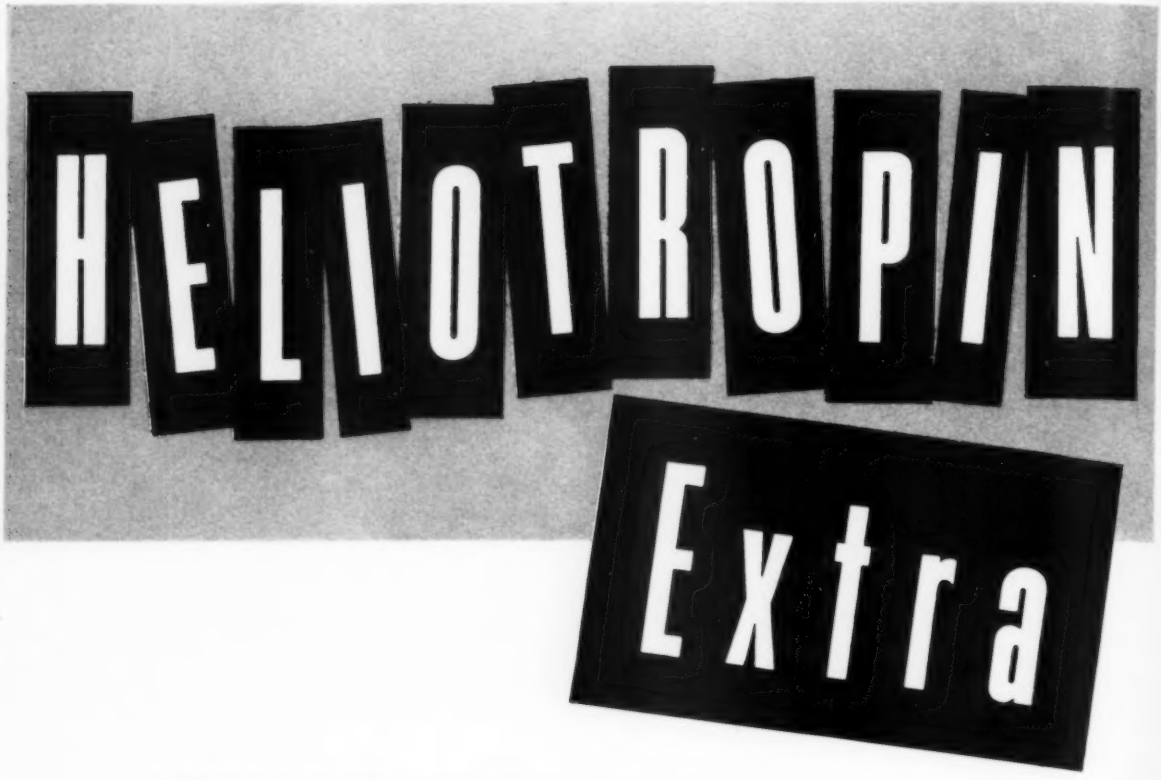

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have we observed a commercial grade of heliotropin comparable to the stable, long-lasting product we are now offering. Produced in fine, sparkling white, consistently uniform crystals, this competitively priced product can best be judged by direct comparison with the usual grades... We urge you to examine and then *try* this really outstanding specialty in your soaps, toiletries and cosmetics... A request by letter, phone or wire will bring you a FREE trial sample of HELIOTROPIN EXTRA by return mail.

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KOLAR

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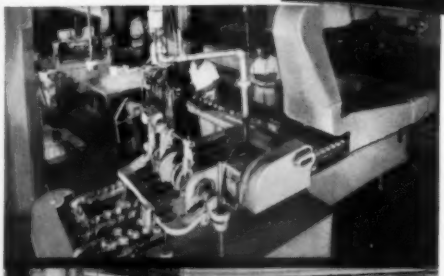

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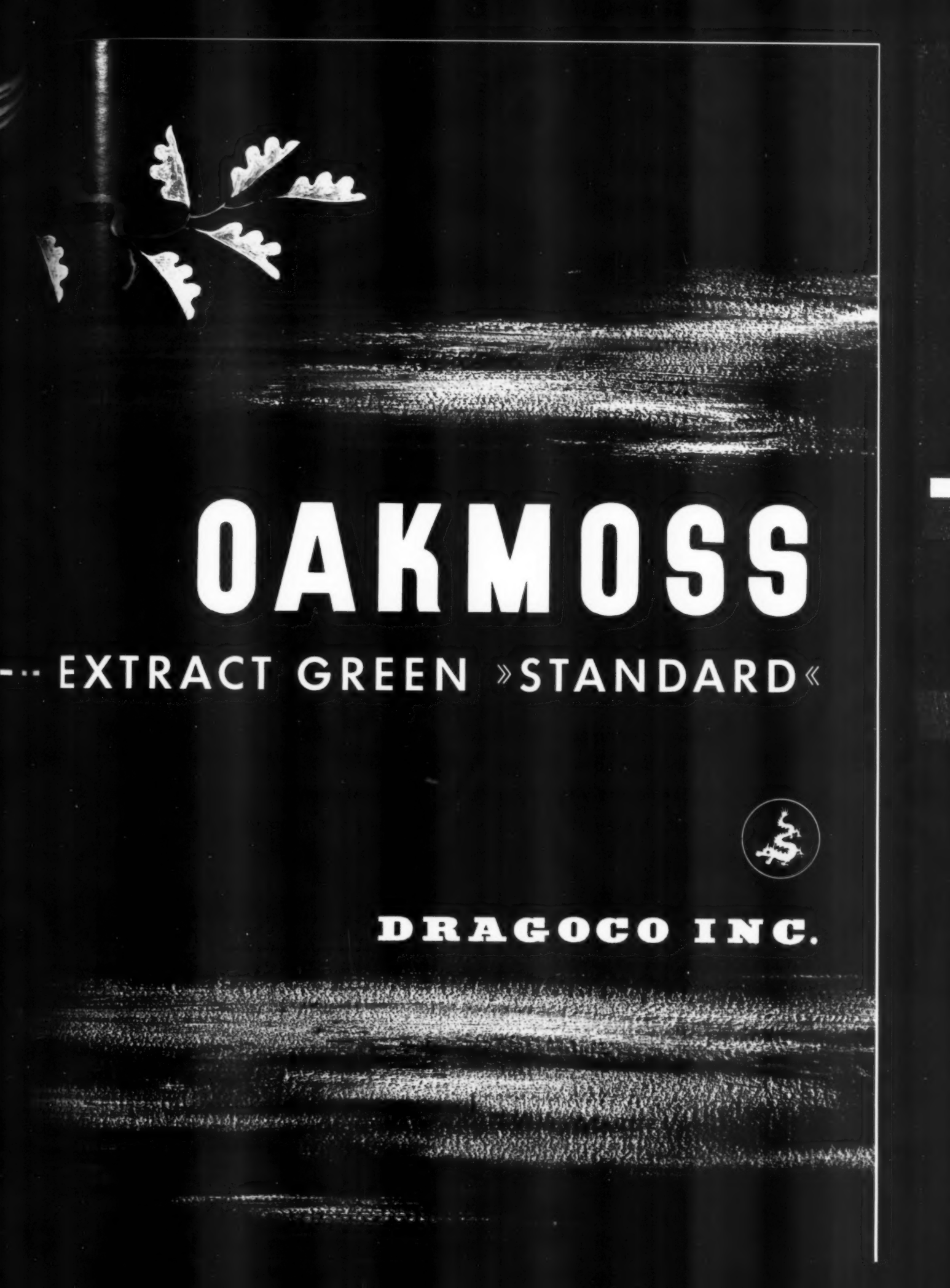
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EXTRACT GREEN »STANDARD«
DRAGOCO INC.

Chemoderms®

...offer documented evidence of low threshold of sensitivity

The individual components that constitute a single perfume compound frequently pose a problem of dermal sensitivity—a problem recognized by dermatologists, physicians, and pharmacologists the world over.

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- **more than 5 years of actual, international consumer use**
- **hundreds of products, used millions of times by men and women**
- **documented and published dermatological reports**

Now, through the use of these carefully prepared, chemically reproducible Chemoderms, you can assure yourself and your customers of dermatologically evaluated perfumes in your cosmetics, pharmaceutical or proprietary products.

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Notes

Did you know that seven Latin American countries have agreed to remove trade restrictions over a twelve year period? They are Argentina, Brazil, Chile, Mexico, Paraguay, Peru and Uruguay . . . . Looks like the Vanilla bean situation is a bit tight for Madagascar and Mexican beans . . . . Evans Research got a patent for putting garden fresh flavor into canned, frozen and dehydrated foods by adding flavor enzymes . . . . Pfizer are now offering dihydroxyacetone in crystalline form . . . . A sunscreen lipstick has been suggested by Bray and Parsons, to protect the lips from burning by solar ultraviolet. The most effective screen used was methyl *p*-dimethyl aminobenzoate mixed with salol according to the authors . . . . Congratulations to Dr. Frank J. Steel who was awarded a Doctor of Science in chemistry degree by Metropolitan University at Glendale, California . . . . My compliments to Bob Horsey on his election to the presidency of the Essential Oil Association. Bob can't get gray on *this* job, if you know what I mean . . . . There is always something to learn it seems. Now there is the Association for Corporate Growth and Diversification. Officers come from McCormick and Co., Inc., Colgate-Palmolive Co., and Chesebrough-Pond's, Inc. It's a national group if you please, publishing its own newsletter THE PERISCOPE . . . . Like so many others, I've had my umpteenth letter starting off "I go to Burpme High School—If you would send me the composition of the best brands of lipstick, it would help me with my term paper (or a new variation—"my project for the Science Fair") . . . . Dr. Bernard (Barney) E. Conley, formerly Director of the A.M.A. Committee on Toxicology, has joined the staff of Hoffman-LaRoche, Inc., as medical research consultant. Congratulations and good luck Barney! . . . . The world loses a great man in the death of Dr. Erich Boehm, who had so much to do with the discovery of the *p*-hydroxybenzoates and the galla antioxidants . . . . To British Revlon's Frank Atkins in his retirement—sunshine and happiness in the early evening of his life . . .Speaking of shampoos, there is a lot more to be heard about the albumin and protein derived surfactants soon. . . .It was a fine series of papers the G.P.A. presented at the Waldorf recently. . . . . . . A polyoxyethylene ether of lauryl alcohol has found use in an acne preparation, along with hexachlorophene and "other ingredients" . . . . You certainly have to compliment Gerry Gray of Wallerstein Labs for his ingenious method of getting people to look at his Rapicidin by sending a select group of prospects a lavish Monopoly game with "plugs" and a sample of his product.

Panax Ginseng

The word Panax is derived from panacea, a cure-all. Other botanical cure-alls are garlic and mistletoe. Ginseng is the wonder drug of the Chinese herbals. Garlic is the drug of the Continental Europeans, whereas mistletoe is the drug of the Druids of Gaul and Britain; most particularly of the Celtic speaking peoples in more modern times.

Ginseng is finding its way into cosmetics presumably as a panacea. Something like 10-20% of either an alcoholic or aqueous extract, equivalent to 20% of the root, is used in such cosmetics. Personally I am anxious to see the clinical proof of effectiveness.

Skin Behavior of Perfumes

Freddy Wells reports quite an interesting (and possibly controversial) series of experiments on the changes and lasting properties of fragrances applied to skin in his SOAP PERFUMERY AND COSMETICS for December. He certainly makes it questionable as to whether it is desirable to apply fragrance to skin as women have always been told to use perfumes and toilet waters—unless the fragrance was pretty well tested out "clinically" to see how it would behave on all or most women's skins.

No one questions the veracity of Freddy's report. But I wonder if what he finds will be just as valid

if he used 200 subjects, women preferably, with no organic ailment known to give skin or body a characteristic odor. Presumably the importance of such variables as whether each hand gave the same reaction; the position of test patch on the hand; the time of day; room temperature and humidity and time of the year were all determined. The tests would then be exactly alike insofar as external influences were concerned. The final variable would be the personal skin biology of the subject on whom the test was performed. Results with single aromatic chemicals might be at variance with mixtures.

I have seen two quite different fragrances encounter a sufficient number of "changes" in odor on people to force the discontinuance of both. In the one case a rather flowery bouquet developed a "urinaceous" character when used in a cream by *some* women. In another case a very refreshing pine lotion used by men developed a strong "body-like-odor". Yet *most* men in this case and the women in the case before, did not so react.

Basically our skins are much alike, comparing men to men, women to women, age to age, etc. But we know too that the skin on one body area is not exactly the same as on another even on the same person. The skin, like other body organs, probably reacts differently at various hours of the day and under diverse influences.

It would be so much simpler if we could run a test on one person or animal and translate that as meaning "on all people".

Freddy Wells' work is thought provocative. There will be yeas and nays. But that is progress. It takes a bit of daring to say things are "so and so" especially in print, particularly if everyone isn't in complete agreement.

Having determined a result, all that needs be done is to prevent it from happening. That is what makes a good perfume (a good perfumer, too).

Freddy, you have done a job that needed being done.

Soybean Cleanser

Although U. S. Patent No. 2,380,830 was granted in 1945 covering the use of residual meals (left after extracting oils from seeds) of particle size between 20 and 60 mesh as a skin cleansing composition when mixed with bicarbonates, phosphates with or without soap, no commercial product of note appeared on the market until the last year or so. Shows you just how long it can be between conception and delivery of the goods.

Another U. S. Patent No. 2,610,153 covers the use of soybean meal residues treated with formaldehyde, adding a detergent and forming into bars, paste or powder.

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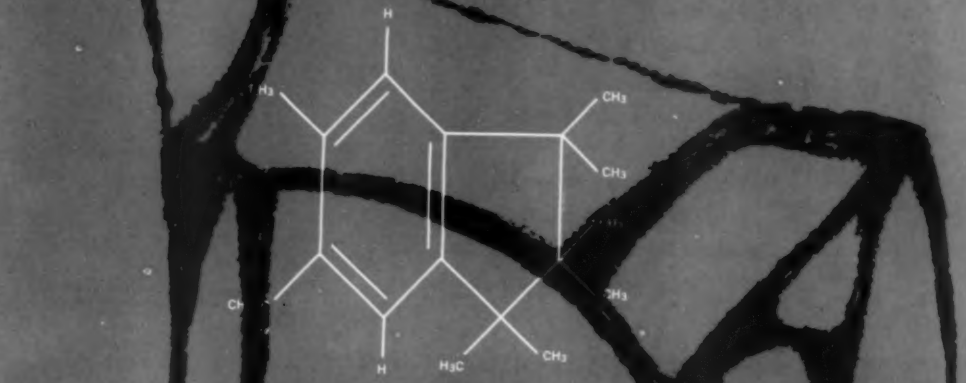

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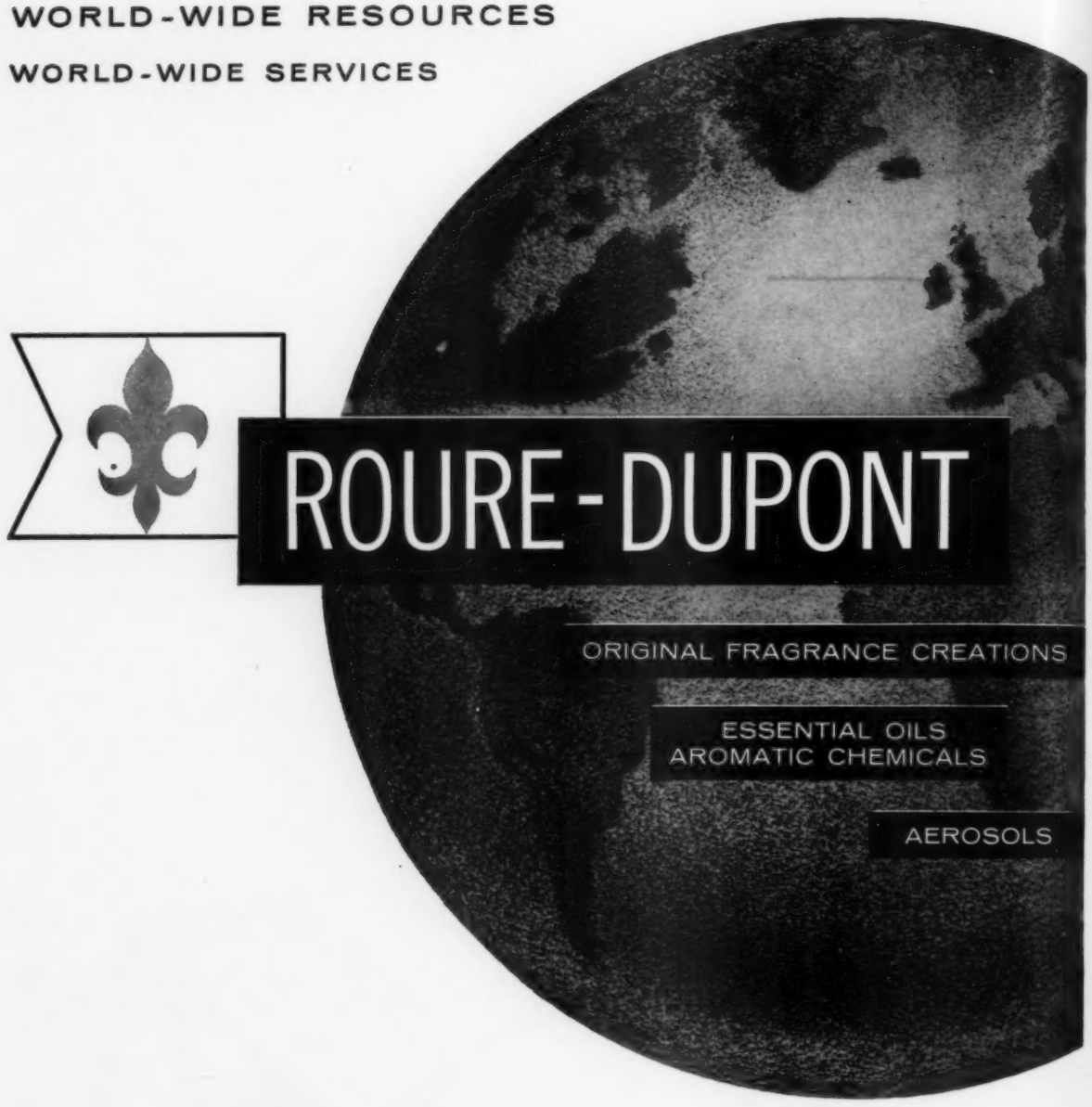

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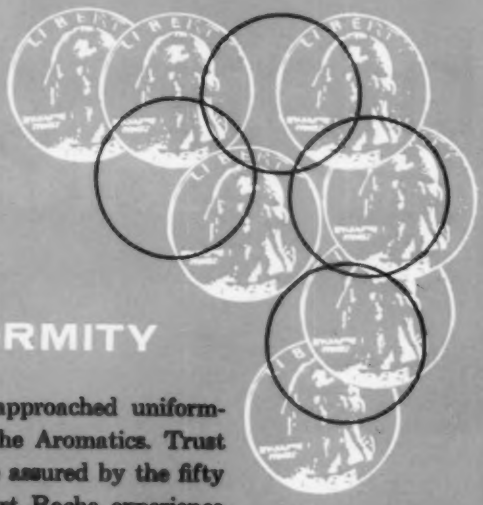

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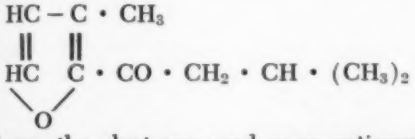
April, 1960 Volume 75—Number 4

Elsholtzia Oil - its application in perfumery

BY DR. HORST SCHMIDT

As early as 1919 Y. Asachina and Y. Murayama distilled and analyzed the oil of the labiated elsholtzia cristata. The investigation carried out at that time resulted in the discovery of a ketone, the elsholtzia ketone, of a 3-methyl-2-isovaleroylfurane with the formula



In Japan the plant was used as an antipyretic and diuretic. Its essential oil was not, however, produced on a commercial scale and consequently did not find any application. Only in recent times has this oil been exported from China so that it would now pay to devote some attention to it.

Recent analytical research on the composition of this interesting oil will show that it consists of a number of different substances, the ingredient responsible for its fragrance presumably being the above-mentioned elsholtzia ketone. Since these investigations have not yet been concluded, it is still too early to publish a report on the details which have been discovered up to the present.

The first odour impression of the oil is a fresh "dry" note with a spicy accent. The odour effect is difficult to define since it cannot be associated with anything found in the usual essential oils. It nevertheless gives the impression of being an oil of "light" odour in contrast to one of a dull type, such as patchouli oil. It is this characteristic fragrance which distinguishes the oil as a "top note".

The characteristic perfume of the white rose is generally produced from a normally formulated rose composition by additions of sandalwood oil, patchouli oil and guaiacwood oil. With the aid of elsholtzia oil the typical note of the white rose can be achieved with remarkable success.

White Rose

320	Hydroxycitral chem. pure
80	Geraniol ex Palmarosa
40	Citronellol L
40	Nerol pure
20	Dimethyl Octanol
120	Phenyl Ethyl Alcohol supra
30	Phenylacetaldehyde Dimethylacetal
10	Citronellyl Acetate
20	Ionone Alpha pure 10%
60-30	Oil of Elsholtzia
15	Oil of Daucus terpeneless 10%
20	Oil of Patchouli terpeneless 10%
10	Oil of Vetiver terpeneless 10%
10	Citral supra 10%
10	Undecylenic Aldehyde 10%
5	Cuminic Aldehyde 5%
40	Guajyl Acetate
20	Oil of Geranium Afric. rect.
40	Abs. Rose de Mai
20	Abs. Cire d'Abeilles supra
900	

The basis of this rose is hydroxycitral, i.e. the chemically pure body and not a fancy composition; the other rose perfumes are normal. The effect of elsholtzia oil is reinforced by small quantities of terpeneless daucus oil, patchouli oil and vetiver oil. This rose

composition can be used to advantage in particular, for the perfuming of cosmetic products and also as a basis for floral and fancy compositions.

The spicy accent of elsholtzia oil suggests its suitability for use in a carnation composition. In this case larger quantities can be used than in rose, due to the extremely strong odour of eugenol and isoeugenol. The spicy character of the carnation is emphasized by additions of terpeneless qualities of bay oil, laurel leaf oil, pimento oil, pepper oil, mace oil and cardamon oil. The floral note is assured by the white rose described above, the quantity of which may of course be considerably increased.

Carnation

300	Isoeugenol
150	Eugenol
90	Oil of Elsholtzia
30	Oil of Bay terpeneless
30	Oil of Pimento terpeneless
20	Oil of Black Pepper
10	Oil of Nutmeg terpeneless
15	Oil of Cardamom
15	Oil of Laurel Leaves terpeneless
440	White Rose
40	Ylang Ylang extra
60	Amyl Salicylate
1200	

The possibility of using elsholtzia oil for flower perfumes is, of course, by no means exhausted by its application in rose and carnation. Very good effects can be achieved, for example, in honeysuckle, jasmin, mignonette, verbena et.

As already briefly mentioned, elsholtzia oil has a "light" note and is therefore particularly suitable for bringing out the top notes in many different compositions. These top notes generally consist of citrus oils, often in their terpeneless form in conjunction with aliphatic aldehydes, the corresponding alcohols and in certain cases their esters. Particularly when using predominantly aldehyde components, without or in conjunction with terpeneless citrus oils, this top note, although fresh at first, may easily give the impression of being somewhat dull and flat. Elsholtzia oil has proved a really excellent base for giving the top note a certain amount of "body". In the following formulae an attempt has been made to obtain a top note from orange oil, terpeneless orange oil, aldehydes: undecylenic aldehyde and undecylicaldehyde, tridecylicaldehyde, isocyclocitral and undecylenic alcohol in conjunction with elsholtzia oil. Additions of spearmint oil, sweet basil oil and shiso oil produce a fascinating note. Depending on the type in which this top note is to be used the proportion of elsholtzia oil may be safely increased.

Top note

450	Oil of Orange sweet Guinea
150	Oil of Orange sweet terpeneless
30	Undecylic Aldehyde 100%
40	Undecylenic Aldehyde 100%
5	Tridecylic Aldehyde 100%
55	Undecylenic Alcohol 100%
60	Oil of Elsholtzia
30	Oil of Spearmint
15	Oil of Shiso Japanese
20	Oil of Basil sweet
15	Isocyclo Citral 10%
10	Undecalactone 10%
120	White Rose
1000	

It will first be shown how an eau de Cologne type can be very nicely produced with the aid of the "top note" described above. The ingredients bergamot oil, orange oil, lemon oil, grapefruit oil etc. are normal. This base is given a wonderful freshness by about 3% of the above "top note". The addition of elsholtzia oil harmonizes remarkably well with the citrus oils used and the amount incorporated can be increased or decreased within wide limits. Depending on costs, the expensive genuine neroli oil may of course be wholly or partly replaced by a first-class synthetic product or used in combination. This mixture gives a very fresh, almost "classical" eau de Cologne. It can easily be perfumed in a very pleasing manner by addition of a fancy note, e.g. to 900 gr of the recipe 100 gr of the extrait oil "blue dream", to be described later.

Eau de Cologne

30	top note
220	Oil of Bergamot
160	Oil of Orange sweet Guinea
160	Oil of Lemon
60	Oil of Grapefruit
60	Oil of Elsholtzia
90	Oil of Neroli big.pet.
60	Oil of Petitgrain terpeneless
30	Oil of Rosemary French
30	Oil of Lavender Barreme
900	

Even more convincing is the use of elsholtzia oil in lavender compositions since the character of English lavender oil can practically be achieved when a large addition is used.

Lavender

300	Oil of Lavender Mt. Blanc
150	Oil of Lavandin terpeneless
150	Oil of Clary Sage Grasse
150	Oil of Elsholtzia
150	Isononyl Acetate
100	Dorisyl (Down)
15	Oil of Spearmint
20	Dihydrocoumarin
10	Methyl Coumarin
20	Resinoid Tonca
55	Exaltolide 10%
1100	

The exaltolide mentioned in the formula may of course be replaced by any modern large-ring musk. For spray application the recipe may be modified without difficulty by a suitable combination with pine-needle oils or their fragrant elements.

The recipe "blue dream" will show how the "top note" described above can be changed into a pure fancy perfume. This perfume is based on large proportions of jasmin, preferably a combination of a

synthetic jasmin of absolute character with a good South French absolute type without too much of a "fatty" odour. The most suitable product for this application would be the so-called Butaflor jasmin of Messrs. Robertet in Grasse, which, as everybody knows, is obtained by extraction with butane gas. This and other butaflors possess the great advantage of being selective extracts since unlike other solvents, such as benzene or gasoline, the liquefied butane gas does not completely absorb the fragrant substances of the blossoms. In certain recipes butaflor jasmin cannot of course replace a normal absolute, but in combination with a synthetic jasmin it offers great advantages since it blends much better. The elsholtzia oil addition harmonizes extremely well with the neroli bigarade oil used. Only a relatively small quantity is required since a small proportion is also introduced into the composition by the "top note". A certain spiciness is achieved by the carnation developed at the start, a little terpeneless mace oil and cinnamon oil Ceylon. As a fixative, a perfumistic ambergris complex is not used, but a product which embodies the true character of genuine ambergris, similar to Firmenich's Grisambrol. The developed base is best blended with the same weight of a jasmin extract, produced by maceration of a South French concrete with alcohol, freezing out and filtering. Extracts of this type have quite a different and superior fragrance to simple solutions of a corresponding quantity of absolute in alcohol and are often the most important factor in the development of the fragrance of a fancy perfume.

Blue Dream

75	top note
55	Oil of Bergamot
45	Oil of Jasmin synth.
20	Jasmin abs. 50%
15	Oil of Neroli big.pet.
35	Oil of Elsholtzia
10	Mimosa abs. 10%
30	Hydroxycitronellal
55	Carnation
5	Oil of Cinnamon Ceylon 10%
15	Oil of Nutmeg terpeneless 50%
60	Iraldeine 100%
30	Oil of Sandalwood East Indian
15	Oil of Vetiver terpeneless
10	Mousse de Chene abs. extra
25	Amber gris synth.
500	Tincture of Jasmin concrete 10%
1000	

Elsholtzia oil is remarkably effective in soap perfumery. Its relatively low price renders it suitable for many applications. It by no means plays the part of a valuable universal filler such as terpineol, but imparts considerable body to the compositions and can easily be blended. It also offers the advantage of emphasizing and strengthening the foundation in a manner which can hardly be achieved with considerably dearer ingredients. Only two examples taken from a large number of possibilities will be described: a floral perfume oil and a fancy composition.

On the same lines as the "white rose" described above, a modification for use in soap perfumery will first be discussed. For the formulation of this composition no further comments are necessary since it conforms with normal practice. The combination of the dimethylacetal of phenylacetaldehyde, benzyl acetate and geranyl acetate with a large addition of elsholtzia oil gives a perfume type resembling the centifolia. This soap oil can, of course, easily be used for cheaper commercial soaps by strengthening the rose alcohols contained therein, increasing the diphenyl oxide and adding a good synthetic geranium oil. For a better quality a certain amount of genuine geranium oil, preferably from an African source, would of course be used.

Rose for Soaps

240	Geraniol pure
80	Citronellol pure
120	Phenyl Ethyl Alcohol
35	Phenyl Acetaldehyde Dimethylacetal
150	Oil of Elsholtzia
15	Benzyl Acetate
15	Geranyl Acetate
10	Cuminic Aldehyde 5%
20	Ionone 100%
10	Nonyl Aldehyde 10%
5	Octyl Aldehyde 10%
20	Rosacetate cryst.
40	Diphenyl Oxide
40	Resinoid Elemi
800	

For a modern fancy soap oil the rose described above may be used as the starting material. It cannot, however, be used alone as the basis of a floral note. For this application additions of alphaamylcinnamicaldehyde, benzyl acetate, hydratropicaldehyde and cyclamenaldehyde or lilial were also used. The use of hydroxycitronellal is of course a much disputed problem. As in all similar cases, much better results are obtained with the aid of Mugoflor H&R which is completely stable in soaps, provided that one does not mind using a speciality. The widely developed wood note is achieved by Dorisyl or a similar brand in conjunction with rectified cedar wood oil, vetiver oil, patchouli oil and calamus oil. Particularly pleasing effects can be obtained by replacing iraldeine with allylionone, which is marketed under the name Cetone V. A small aldehyde addition consisting of undecylenic aldehyde and methyl nonyl acetaldehyde is unavoidable in order to preserve the character of this composition. In view of the somewhat strongly coloured resin fixatives, and also considering the small quantity of carnation, heliotropin and musk ambrette, this perfume oil is of course unsuitable for pure white soaps, although a slight pink or blue shade enables it to be used.

Only a small selection can of course be offered within the scope of this report in order to give the specialist an idea of possible applications. Since this oil is now being marketed for the first time on a commercial scale no experience is available up to the present, at least as far as information in the technical press is concerned.

When further work has been completed with this

Fancy Bouqet for soaps

220	Rose for soaps
60	Terpineol chem. pure
30	Alpha Amyl Cinnamic Aldehyde
30	Benzyl Acetate
10	Hydratropic Aldehyde
10	Cyclamen Aldehyde
20	Hydrocycitronellal
40	Cinnamic Alcohol
140	Dorisyl (Down)
55	Oil of Elsholtzia
30	Oil of Cedarwood rect.
15	Oil of Vetiver
10	Oil of Patchouli
10	Oil of Calamus terpeneless
30	Iraldeine 100%
10	Methyl Nonyl Acetaldehyde 10%
10	Undecylenic Aldehyde 50%
10	Carnation
20	Oil of Ylang Ylang II
10	Resinoid Olibanum
10	Resinoid Labdanum
10	Resinoid Tolu
30	Resinoid Storax
10	Mousse for soaps decolorized
30	Coumarin
10	Heliotropin
20	Musk Xylol
10	Musk Ambrette
900	

interesting and strong natural perfume a number of other applications will no doubt be found and the author feels sure that they will prove even better than the first impressions which he has published.

On the whole, the use of fairly large quantities has been suggested, whereas small to smallest additions would doubtless lead to surprising effects when used in different compositions, particularly in very highclass ones. Since the exact chemical composition of the oil has not yet been determined, it will doubtless be refined by chemical treatments after conclusion of the research work, and the way thus opened for many new fields of application. At all events, the present impression leads to the conclusion that a thorough investigation of this interesting raw material in the perfumery field should be carried out.

Why Color Matchers Differ

An item appearing originally in Textile World, and reprinted in American Dyestuff Reporter for October 5, 1959.

Recent experiments conducted by the National Bureau of Standards give clues as to why experienced color matchers see the same shade differently.

The purpose of the tests was to define the color response of the average human eye. The physical properties of color standards can be measured by strictly physical methods; however, to compute a valid numerical specification of an observed color, the eye's response to that color must be determined.

Tests were made by means of plaques colored gray by two methods. A simplex gray was produced by mixing black and white pigments. A complex gray was made by mixing yellow, green, purple and white pigments.

When a person of normal vision views these plaques so that the samples subtend an angle of 10° in daylight, the simplex gray appears redder than the complex gray. However, at a distance from which the samples subtend an angle of 2°, the simplex gray appears greener than the other.

This phenomenon occurs because the retinal image of the samples at the 10° position covers both the macula and a large portion of the retina, while at the 2° position the retinal image falls wholly within the eye's macula, which is yellow. The effect is similar to placing a yellow filter in front of the eye for the 10° position.

Consequently, the 10°-field results depend primarily on lens pigmentation, and the 2°-field results depend not only on the lens but to a large extent on macular pigmentation.

The age of the observer was found to influence his color response in this test. Thus, the youngest observers described the simplex gray as redder than the complex gray at both the 10° and 2° positions. By contrast, the oldest observers saw the simplex gray as greener in both positions.

Consequently, when color matchers disagree on a match, the angle of view and the differences in age must be taken into consideration.

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Biological tests demonstrating the activity and cosmetic applications of the total human placenta

BY MADEMOISELLE ANNE-MARIE BUREAU
Institute Merieux

The nature of the active principles of the total placenta extract lyophilised remains unknown even though a few of their phsico-chemical properties have been determined. But it is possible to demonstrate the activity of the total Placenta extract lyophilised by biological tests as well as by the experimental and therapeutic results.

I.-Tests in vitro.

a-*Phyto-braking action*: At some relatively important concentrations one can observe clearly a braking action on the growth of the stems, in the culture of lentil seeds (2).

b-*Germ culture*: If placenta extracts are added to the cultures of certain lactic bacteria, the production of lactic acid increases. Meyer and his collaborators (24) using certain selected micro-organisms have been able to establish a direct relationship between the bacterial metabolism and the concentration in biostimulines of certain organ extracts. The same author has also observed (25) a decrease of the activity of certain antibiotics when in the presence of biostimuline extracts of the Filatov type.

Variel and Choay (26), using organ extracts, have isolated a fraction with a biostimulating activity which favors the growth of micro-organisms.

c-*Growth of embryos from triton eggs*s Culture of the eggs starting from the undivided stage or the blastula stage in mixtures with or without placenta extracts; in the presence of placenta extracts, the results are very clear: slight delay of the development on the individuals without caudal bud in the case of the young embryos obtained which present also a certain degree of spina, bifida, that is to say appearance of abnormal forms, much later (27).

d-*Culture of tissues*: This technic consists in having the explants grow on a support composed of coagulated rabbit plasma in the presence of a nutritive liquid. The culture of the tissues takes place in fixed boxes or in roller-tubes, and is maintained at 37°C. The reading is made on the fourth day. The calculation of the growth index of the cultures activated by the organ extract, compared with the index of the cultures without organ extract, gives the value of the cytopoietic power of the total placenta extract.

II-Tests in vivo.

a-*Cytopoietic action*. Tadpoles are placed in crystallisers with insufficient nutrition. Organ extracts are added activating the growth, with a very noticeable reduction of the mortality and acceleration of the development, metamorphosis and regeneration of the tail aftersection.

b-*Corticostimulating action*: It is determined on human beings (28). This phenomenon is interpreted by a positive Thorn test and the humoral modifications forseen. We have verified this action on the guinea-pig treated with placenta extracts; the Sayers test, used under these conditions, has constantly been positive. Besides we have isolated from the placenta

tissue a preparation of adreno-corticotrope as indicated above (23).

c-*Cicatrizing action*: We have determined this on human beings (necrotical capillarites, leg ulcer) treated by applications of total placenta extract either externally (solution or pomade) or internally (injection) (29-30).

The speed and the importance of healing are measured following the Lecomte du Nouy technic, that is to say by taking a tracing of the cutaneous surface with a cellophane paper and measuring the tracing. The results expressed in graphic curves show a gain of time of more than 50% in comparison with the wounds treated in the usual way and the action on the extra scar tissue forming is particularly powerful, making it necessary to slow this extraordinary proliferation.

Other authors have also observed this important action on cicatrization, particularly Nakamura (31) who experimented on animals.

d-*General stimulating action*: Ischlontsky (32-33) has found a stimulating action of the extracts of organs on the hormone secretion in animals, and the regeneration of senile animals.

Elderly me injected with total placenta extract Lyophilised showed a return to normal in the ratio of the rate of blood Glutathione to the number of red globules. This rate, as shown by Binet, rises in an elderly man, therefore this therapy is particularly active in the cases of senescence (34).

e-*Anti-inflammatory action*: Romani (35) utilising the technic of experimental oedema of the rat, has ascertained its absence in animals treated with a preparation of organ extracts.

f-*Anti-infectious action*: In animals experimentally infected, Vairel and Choay (26) have verified after action by the same preparation, not only a raise of the rate of formation of gamma globulines but also an important increase in resistance to infection.

g-*Anti-toxic action*: Ratsimamanga (7) experimenting with a principle drawn from miscellaneous organ extracts, uses its anti-toxic activity against hepatic poisoning by carbon tetrachloride. He is the first author to have made a precise study of the active constituents which he connects, as we have already mentioned, with synergistic ensemble of amino-acids.

COSMETIC APPLICATIONS OF ORGAN EXTRACTS

The therapeutic action of total organ extracts is essentially "aspecific" in that it acts upon the entire organism, tending to enhance its natural process.

The cosmetic uses of these extracts are thus extremely varied but they always have as a goal the improvement of tissue trophicity, vitality, and consequently of their aesthetic aspect.

I. BUST

A steady application on the bust, except during the cathamenial periods, of total extract reconstituted, rapidly bring a noticeable development of the breasts.

Beneficial effects are obtained by spreading a cosmetic preparation (cream or liquid cream) containing from 30 to 50% of total extract reconstituted, or by spreading a total extract reconstituted and diluted at 1:10 in proper solvent (glycerol: 1 part; propylene glycol: 1 part; distilled water: 2 parts) This application will be followed by a light massage. The action of the total placenta extract will be the fastest when the preparation (cream) is the richest in extract and the most penetrating.

II. PUFFINESS, SMALL WRINKLES, CROWS FEET, RINGS AROUND THE EYES AND DOUBLE CHIN.

A daily application, after a deep cleansing of the skin, of total extract reconstituted, pure or diluted at 1:10 in physiological serum, (isotonic sodium chloride solution) will improve the appearance of small wrinkles and crows feet; double chins become less noticeable.

A non-ionic cream containing high concentration of human placenta extract reconstituted but without any allergenic component can be applied to the eye area, to attenuate the unaesthetical effect of circles and the dark rings under the eyes.

III. SENESCENCE: Dried up, flabby, pigmented, etc., skins.

In the case of an accentuated senescence, one can use applications of human placenta extracts, reconstituted, on all the face using an impregnated gauze and leaving it for 10 to 15 minutes on the skin after having cleansed the face thoroughly. After removing the gauze, follow with light massage.

IV. REJUVENATING MASK, RELAXING AND DECONGESTIC MASK, WEIGHT LOSING CURE-

The mask obtained from human placenta extracts reconstituted can be used in many other cases, such as:

a-When after too violent an exposure in the sun, the wind, cold weather, the skin of the face is irritated or congested, one or two applications in a beauty shop or at home, are enough to rapidly improve this condition.

b-In the case of rejuvenating the appearance of the face or lightening of the skin. The treatment must go together with a relaxing treatment, general balneotherapy, a diet, based on a general medical check-up.

c-A tired face can be cleared by daily applications (but not at the time of the cathamenial periods) of gauze impregnated with total placenta extracts reconstituted.

V. CELLULITS

The physiologic phenomenons which are gathered under the general term "cellulitis" must be the object of a very careful medical examination. When the treatment prescribes "massages", one can use, at the time of the massages, cosmetic preparations (massage creams, lotions) which contain strong proportions of human placenta extract reconstituted. The massage can take place after a sudation (steam) treatment

or other usual technics, but it will have as its aim, to favor the elimination of a certain number of products of katabolism, which will bring an abundant "diuress".

VI. EVENING MAKE UP, INVISIBLE MASK

The reconstituted placenta extract (dilution in the physiologic serum) enables one to realize an evening make-up, that is light and supple. One must apply this extract, with a cotton pad, on a carefully cleaned skin. When drying, the micro-layer of extract leaves on the face a supple film on which one can apply the make-up. This invisible mask gives to the face a silky look and allows a light tension of the tissues which "shades off" small imperfections.

VII. UNAESTHETIC SCARS

A vaso-motor massage, or rubefacient, followed by an application of human placenta extract reconstituted, can achieve a long and efficient "cure" of unaesthetic scars. The massage has a mobilizating action which favors blood irrigation and the slow transformation of the cheloides; placenta extract has a very definite action on the reconstitution of the tissues.

This is how the scars (after an accident) which very often take away the beauty of a face, can be attenuated. Even though the treatment may be long and may require much tenacity and patience, it is without any doubt, one of the most efficient, mostly in the case of scars of the face.

VIII. DAILY CARES OF THE FACE:

The daily care of the skin must vary according to the state of the skin.

-In the case of young skins, a light massage at night, before going to bed with a cream rich in placenta extract, or with reconstituted extract, will be sufficient.

-In the case of damaged skin (through atmospheric condition—sun, wind, cold) or in the case of over tired skin: 2 applications daily, one in the morning and one at night, are necessary.

-For more effective results, it is necessary to use a cream in the morning and at night and to apply the extract under it in the form of a film, every evening preferably or at least three times a week.

IX. CARE OF THE HANDS

The use of cream containing placenta extract (reconstituted) containing some fat and humectant substances, will allow the hands to keep their beauty. The application of placenta cream to the hands in the evening normally follows the application of placenta to the face.

X. TOILET WATER

Toilet waters containing humectants and alcohol as a base, as well as after shaving lotions, can also be enriched with placenta extract (reconstituted).

XI. TOILET SOAP

Mild toilet soap enriched with lanoline or with glycerol, are excellent extract-carriers for total placenta. Their use can bring to the skin substances which favor skin vitality even though present momentarily.

XII. HAIR PRODUCTS

Hair treatments (baldness, fat seborrhea, irritation of the scalp) can be improved with the help of placenta extracts reconstituted or diluted at 1:10 in a physiological serum (isotonic sodium chloride solution).

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Editor's Note: This article was obtained as a result of many inquiries concerning biological tests used in determining the activity of placenta extracts. After reading the original manuscript a number of questions occurred to the editor. These were forwarded to the author and point by point answers appear below.

We are replying, point by point, to your questionnaire on the lyophilized human placenta extract.

1. *Physical and chemical incompatibilities*: Action of heat, irradiation by ultra-violet or infra-red, and ozone adversely affect the activity of placenta extract. Moreover, a pH in excess of 10 or lower than 3.5, constitute limits which should not be exceeded.

2. *Can lyophilic placenta extract, reconstituted in flasks of 50 cm3, be kept without loss of activity, if it is used, daily, that is, exposed to air, and in contact with human skin?* Obviously, this would depend on the period of exposure, the surrounding hygienic conditions, and the presence or absence of antiseptics in the placenta extract. However, it is necessary to know that the reconstituted placenta extract represents a particularly potent culture medium, which will mean undeniable microbial or fungus growths, after even a short contact with air, if there isn't an effective preservative in the medium. This is the whole problem.

3. *What is the best and most active antiseptic for the reconstituted extract?*

We have carried out a certain number of studies which have not yet been published (Mr. Trouyez). These studies show that acid antiseptics (benzoic and sorbic acids) produce a substantial deposit. This deposit must be due to the instability of these types of products in a relatively neutral medium. On the other hand, the para-hydroxy benzoates and thimerosol, are the best antiseptic agents. They do not affect the alkaline phosphatases of the reconstituted extracts preserved at 4°C. This stability of the alkaline phosphatases with antiseptics is being determined at higher laboratory temperatures, and for a period of several months. This stability will be satisfactory at

ordinary temperatures. It should be noted that the use of para-hydroxy benzoates may limit the choice of excipients, because of the inter-action between the para-hydroxy benzoates and bodies such as the Tweens.

4. *Interaction of Ethyl Alcohol and Essential Oils on the stability of extracts.*

Tests are now in progress on this problem. Their particular purpose is to set the maximum percentage of absolute alcohol compatible with the total reconstituted extract.

5. *The physiological serum, in France, designates the isotonic solution of Sodium Chloride at 8.5% Grs.*

6. *Minimum concentration of extract to have an active product? Is there a maximum?*

It seems to us that a local preparation should contain at the minimum 25% to 30% of a total lyophilic extract, which is then diluted at 10%. There is no maximum, since the total lyophilic extract can be reconstituted at its organ weight, and used as such on wounds as a bandage.

7. *Complex composition of the total Placenta extract.*

We are not able to supply any exact details on the various questions brought up and the various active principles mentioned. However that be, this concerns a total organ extract which must contain the steroids usually present in the Placenta. However, we can affirm the presence of a cortico-stimulating substance, having the same activity as A.C.T.H. which has been isolated in the Placenta.

This substance was found to be active "in vivo" in animals or in humans in the same conditions as A.C.T.H.

8. *Action of total lyophilic extract on the excretion of 17-keto steroids.*

Tissulary therapeutics in man has always been taken as a cortico-stimulating therapeutics, that is, making the Thorn test vary in a positive manner. However, this Thorn test has been explored only on the eosinofil side, and the side of connection: uric acid/creatinin. (Extract from "Societe de Dermatologie et Syphilligraphie", April 8, 1951 meeting, Page 585).

Additional research, not carried out directly by us, has shown the elevation of the excretion of the 17-keto steroids under the influence of an *injectible* placenta extract.

9. *Is the human Placenta better than the animal Placenta?*

We think that the human placenta extracts, from the cutaneous angle, present advantages over the placenta extracts of bovines. This does not concern extracts of the Filatov type, that is, those obtained after denaturation by heat, but rather total organ extracts, consequently bringing in non-modified protein substances. On this subject, the human placenta to us, appears superior, because it doesn't run the risk of bringing about sensitizations of the anaphylactic type, whether they be humoral or cutaneous.

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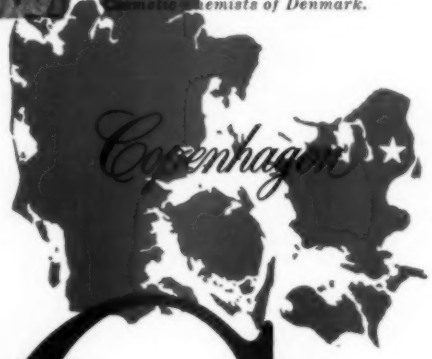
SPEAKING FROM DENMARK

ERIK THOMSEN *is a native of Denmark. Since graduation in 1947 he has been employed with Danish soap and cosmetic firms. He is now manager of development and production with Breinings Laboratorium (Madsen and Wiese A/S), one of the oldest Danish cosmetic firms. He is present secretary of the Society of Cosmetic Chemists of Denmark.*



SPEAKING FROM SWITZERLAND

DR. R. HEILINGOTTER *is a graduate of the University of Gottingen, Vienna. He has specialized in hair-waving and hair-dyeing cosmetics since 1932. In recent years he has been chief chemist in a Netherlands plant and now practices as a scientist on hair-cosmetic researches at St. Blaise/Neuchatel, Switzerland.*

REVIEW OF Glycerine IN HAIR PREPARATIONS

SHAMPOOS, RINSES, LOTIONS AND TONICS, RECONDITIONERS, FIXATIVES AND LACQUERS, DRESSINGS

by Erik Thomsen

SHAMPOOS. It is always desirable that shampooing be accomplished in such a way that the resulting hair is not left in a condition of fluffiness, caused by lack of adherence or static electricity. As the demand of the buying public is still for a good foaming shampoo, it has—as far as I have seen—not yet been possible to create a shampoo to be sold completely without that downy fly-away hair drawback. In any event, glycerine is considered by some to be a helpful aid. Powers mentions, "Glycerol helps retain moisture and delay evaporation. Addition in shampoo formulations keeps the hair softer and helps moisture retention." For other reasons, I find glycerine of great value as a shampoo ingredient. It can act as an aid in manufacture, as a clarifier or a bodying agent. I have to admit, that my experience regarding soap shampoos is of little value, because we in Denmark have very hard water, rendering the soap type shampoo unsatisfactory and not very popular. For the manufacture of liquid soaps, however, I know how useful glycerine is. If it is not already present in the product as a result of saponification of fats, it simply may be added. Practically all formulas for soap shampoos in the literature contain glyc-

(Continued on Page 2)

HEAT-WAVING, DYEING, REMOVING AND STRAIGHTENING

by Dr. R. Heilingotter

HEAT-WAVING. The incorporation of "oily" substances into heat-waving lotions is considered beneficial to the hair. In order to minimize hair damage by the action of heat and the alkalinity of heat-waving lotions and to overcome hair dryness, these preparations are often provided with suitable additions of glycerine, especially to those which are applied at higher temperatures. According to Sarensen (1) an addition of half per cent glycerine, especially for difficult to handle, coarse hair, is suitable as a softening medium:

Sodium sulfite	10	parts
Ammonium carbonate	8	"
Ammonia 26%	2	"
Glycerine	0.5	"
Water, to make	100	"

Glycerine serves also to give a desirable lustre to the finish of the hair after it has been treated with the permanent-waving liquid. For the same reason Janistyn (2) suggests the following formula for a heat-waving lotion:

(Continued on Page 3)

erine, which illustrates the necessity of its presence as a clarifier (solubilizer), an antifreeze, a thickener, perhaps a foam stabilizer—as well as a hair conditioning agent.

What has been said above regarding soap shampoos is valid also for soapless shampoos; there will always be a reason for the addition of glycerine to a shampoo. I have in my own practice successfully used it as a bodying agent and an antifreeze in a liquid shampoo for hair-dressers, and in a cream shampoo based on Sodium laurylsulphate (in an amount of circa 5%) to prevent granulation of the cream during storage.

RINSES. In the days of soap shampoo, a hair rinse was simply a solution of a weak organic acid, used to dissolve the deposit of lime soap that would otherwise leave the hair dull and without lustre. Today a hair rinse is used to bring about better setting properties of the hair, to prevent fly-away after-shampoo effect, and eventually bring about brilliance to the hair. To promote that, a modern rinse will have to contain a cationic agent, a humectant and some oily substance. It may be presented to the customer as a one-use-pack or in larger bottles. It will appeal most to the buyer, if it has a creamy appearance, looking more "nourishing" than the plain clear solution. It may be provided with a suitable dyestuff, making a color rinse. Here is a simple emulsion to be used as a starting point for further development:

HAIR RINSE

Ethoxy lauryl ether	5	Glycerine	4
Heavy white oil	16	Ethanol 96%	16
Laurylpyridiniumchloride	0.2	Water	58
Polyvinylpyrrolidone	0.8	Perfume to suit	

The mixed oil and emulsifier are stirred during slow addition of the water. When the emulsion is finished, add the rest, and finally the alcohol.

LOTIONS AND TONICS. The same role is played by glycerine in alcoholic lotions in building a moist film around each hair shaft. Here partial dehydration is often brought about by the plain alcoholic solutions of perfume. A humectant helps eliminate this. The amount of glycerine in alcoholic hair lotions (if containing vegetable extracts or drugs generally called tonics) may vary from 2-12%, increasing with the water content. If kept at a reasonable ratio to the water (not over 1:4) in the lotion there is no risk of making a too heavy and slowly penetrating preparation. Glycerine will make the hair soft, supple and brilliant. In certain cases it even will help in keeping the perfume in solution and the lotion crystal clear.

HAIR RECONDITIONING CREAMS. The defatting action of the modern shampoos and the more and more frequent practice of dyeing and waving treatments with serious consequences to the hair and the scalp have resulted in a still increasing demand for preparations with the capacity of restoring the hair qualities lost during the mentioned proceedings, as for instance lustre, suppleness and softness.

Usually reconditioning creams contain lubricants (petrolatum, mineral oil, fatty esters, etc.), body giving substances (fatty alcohols, waxes, spermaceti), nutrients (lanolin, lecithin, hair hydrolysates, linoleic acid), emulsifiers, and humectants such as glycerine. The following formula for a hair reconditioning cream, which is very easy to make, simply by stirring the two phases together at a temperature of 70° C, perfume, cool and pack, gives a rather stiff cream, which the hairdresser must dilute with water to a semifluid emulsion before applying it to the hair after shampooing or permanent waving.

Cetostearyl alcohol	20	Sodium laurylethoxy-sulphate	1.5
Hydrogenated sperm oil	2	Glycerine	5
Soya lecithin, extra light	1	Boric acid	0.5
White oil	3	Polyvinylpyrrolidone	0.5
Isopropyl linoleate	2	Water to make	100

Perfume, colour, preservatives q.s.

As the cream is intended to be rinsed out of the hair after having been in the hair as a treatment for about 15 minutes, the glycerine content is here kept as low as 7% of the water phase. At that level it is believed to have a good chance of penetrating into the hair shaft, remaining there after rinsing and exercising a softening, swelling and humectant action in the hair. Using too high a content of glycerine will result in reduced penetration.

Besides this "treatment" property of glycerine, it also adds to the keeping properties of the cream—which, as it will be used in hair dressing salons, probably will be packed in jars, and consequently be prone to drying.

FIXATIVES AND LACQUERS. The purpose of these two types of non-greasy hair dressings is the same, the difference principally based upon their way of application. While hair fixatives are applied to the hair before—and aiding in—the setting, the lacquers are sprayed onto the hair after the coiffure has been finished. Colloid substances are in both cases used as binders and film formers for the hair. In fixatives they are mostly gums, in hair lacquers shellac or resins, and as the hair lacquers must be sprayed, they must be of low viscosity, while the fixatives are of the gel-type or high viscous lotions.

Gums, shellac and resins are usually rather brittle substances, which have to be plasticized to avoid the unpleasant flaking of the film, which in bad cases looks like dandruff. Here again we can take profit with glycerine: as a plasticizer and softener for the film-building substance, as a conditioner for the hair, so it looks more supple and is easier to set, and as an aid in redissolving the film, either for resetting of the hair with a moist comb or in a desired removing of the film.

The hair fixative is a very nice semi-gel, easy to apply and really corresponding to its name of fixative:

High viscosity sodium alginate	9	Ethanol 96%	15
Glycerine	3.2	Perfume (water soluble), colour preservatives	q.s
Water, to make	100		

Disperse the alginate in alcohol and glycerine. Add the water and when smooth, the perfume. It is most convenient to fill it into wide necked bottles immediately after the mixing, as it gets pretty viscous in a couple of hours.

DRESSINGS. In the last decade, the old fashioned liquid brilliantines (consisting mainly of vegetable and mineral oil) have been outclassed by the cream-type hair dressings, principally manufactured as the well known borax-cold creams, the difference being a lower melting point of the wax-oil mixture and eventually addition of further emulsifiers. Such creams are virtually mixed-phase emulsions, with a tendency to drying when exposed to the atmosphere, so a glycerine addition will be of benefit for keeping qualities. Further it will be of aid in the manufacture of oil in water creams, manufactured with the modern synthetic polyglycol ether-emulsifiers.

Here is a formula for an oil-in-water cream, easy to spread, giving a nice lustre to the hair without being too greasy. The glycerine will, together with the emulsifiers, help re-emulsification of the fatty substances during shampooing, an advantage which is often missing in hair creams

NOTE: Inclusion of formulations in this series of articles does not constitute a recommendation by G.P.A.—nor warrant that they are free from patent or other legal restrictions.

and almost always absent in liquid brilliantines. The cream is suitable for packing into tubes, but because of the nonionic emulsifier, the preservative must be selected very carefully.

HAIR CREAM (dressing, grooming cream)

Stearic acid	6	Isopropyl myristate	12
Spermaceti	6	Light white oil	20
Glyceryl monostearate	2	Triethanolamine	0.8
Ethoxy lauryl ether	2	Glycerine	4.2
White petrolatum	6	Water, to make	100
Lanolin	2	Perfume, preservative	q.s.

Melt together the waxes and oils; mix the water phase. When both phases at a temperature of 70°, gradually pour water phase into oil-phase, while stirring slowly. Continue stirring till cool; add perfume at ca. 45°C.

(Continued from Page 1)

HEAT-WAVING, DYEING, REMOVING AND STRAIGHTENING
by Dr R. Heilingotter

Ammonium sulfite, 22° Be	1.0	Triisopropanol aminoleate, 25%	2.0
Ammonia, 0.910	20.0	Nekal	0.1
Glycerine 28° Be	2.0	Water, dist.	73.0
Polyethylene glycol	2.0		

The addition of glycerine to cold-wave lotions has not been found as necessary because of the self-softening action of the active ingredients of such solutions, but some home-wave lotions contain thioglycerine as active agent in alkaline medium. Glycerine, in this case, is used as raw material for the synthesis of the thio compound.

Recently, weakly acid or nearly neutral cold-wave lotions have been introduced. One of these preparations (3) uses thioglycerine without any addition of ammonia or another alkali, at a ph of 4 to 6. Thioglycerine can be prepared from glycerine-glycid (epichlorhydrin) and hydrogen sulfide or barium sulfide, according to Smith and Sjoeberg (4) or by Morell (5) from a mono-chlorhydrin with potassium sulfide or hydrogen sulfide.

Acid cold-wave lotions may also consist of mercaptan carbonic esters, containing at least one hydroxyl group in the molecule, at a ph of 4 to 7, according to Richter (6). In this case glycerine-monothioglycolic ester and/or similar other esters are used and glycerine is necessary as raw material to produce this compound.

A completely different way to produce heat-permanent waves eliminates electrical energy to obtain the curling effect together with usual alkaline heat-wave lotions by replacement of the electrical heat by chemical heat. Use is made of combinations of chemicals to give sufficient heat by chemical exothermal reaction, if moistened. Besides other combinations an oxidative reaction between permanganates and glycerine or other oxidizable substances, has been found as particularly effective. This has been described by Bonat (7) and Francis (8).

HAIR DYEING. Glycerine is often used in hair-dyeing preparations, for example, in a "Henna liquid shampoo" described by Jannaway (10):

Soapless shampoo base	20 to 30%	Lawsone, synthetic	0.08 %
Lactic acid	0.5 %	Alcohol	10.0 %
Glycerine	5.0 %	Water, to make	100 %

Glycerine has been of special importance to the manufacture of metallic hair-dyes. An addition of glycerine to hair restorers is usual to keep them humid during application. Creamy preparations of this kind are intended to provide an additional amount of glycerine, according to Jannaway (11):

Emulsifying wax	10 g.	10 g.	Copper chloride	0.5 g.	—
Cetostearyl alcohol	5 g.	5 g.	Lawsone, synthetic	0.02g.	—
Liquid paraffin	8 g.	— g.	Indigo, soluble	0.1	—
Glycerine	5 g.	6 g.	Water, dist.	68.0	75 g.
Lead acetate	3.4 g.	4 g.			

Modern oxidation hair-dyes are today mostly manufactured as cream or jelly in a consistency suitable for tube filling. The cream hair-dyes have been the first of this type and have been introduced on the market by Stroher (12). As a cream base, glyceryl monostearate has been used and many imitations utilize the same emulsifier, alone, or in combination with other fatty compounds able to form an emulsion. Glycerine is necessary to produce glyceryl monostearate. This emulsifier is not only in use to prepare cream oxidation hair-dyes but also to produce cream color rinses.

Liquid color rinses, which have the same duty, to give a light coloring effect to the hair, are prepared with addition of glycerine, conforming to a formula, given by Jannaway (13):

Chamomile extract	12.0 g.	Glycerine	2.5 g.
Rhubarb, conc. infusion	3.0 g.	Chamomile oil or compound	0.5 g.
Lawsone, solution 1%	2.0 g.	Water	80.0 g.

HAIR REMOVING. Glycerine is also used in depilatory preparations in appreciable amounts; they are mostly offered as cream products. Glycerine gives softness and suppleness, preventing drying during application to the skin. In general depilatories are not declared skin compatible preparations since they attack not only the hair, but also the skin, making it rough and brittle. Glycerine has to counteract this disagreeable, secondary effect. The following formulas, according to Winter (14), show that glycerine is useful in considerable amounts:

Strontium sulfide	30	27	Lanolin, anhydr.	—	8
Calcium sulfide	—	3	Glycerine	10	8
Calcium carbonate	15	—	Water	30	25
Gum tragacanth	—	0.3			
Starch	15	5		100	76.3

Also in the modern cream depilatories based on thioglycolates glycerine is used in appreciable quantities, as the formula of Keithler (15) shows:

Calcium thioglycolate	6%	Sea Kem Type 11	2%
Carbowax 6000	9%	Alcohol	5%
Glycerine	10%	Water	47%
Zinc oxide	20%	Perfume	1%

HAIR STRAIGHTENING. It is a fact that individuals with straight hair want curly hair and the individuals with curly hair believe straight hair to be more desirable. But to the great percentage of individuals with natural curly hair, hair straighteners have become today more and more important. An ancient method was that of plastering down the hair by the use of gums or resinous-fatty vehicles. Later on an alkali type of straightener came into use; these formulas contain glycerine:

Sodium hydroxide	4	10	Oleic acid	—	5
Glycerinmonostearate	16	—	Glycerine	5	5
Stearic acid	—	15	Water	75	65

In modern preparations, thioglycolates have been used for hair straightening. Such preparations contain 1 to 12 per cent thioglycolic acid; sufficient alkali, for example ammonia to form ammonium thioglycolate; an organic amine, such as ethanolamine to produce sufficient alkalinity; ingredients able to give viscosity and adhesion, such as alkylcellulose, alginates, polyvinyl alcohol, Carbowax and sultable other products (16). Another suggestion by Morelle (17) uses thiolactic acid together with a water soluble non-toxic alkali, jelly forming compounds and water. In both formulas an addition of glycerine seems to be desirable.

Bibliographies available upon request.

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Useful Combinations of Anionic and Cationic Surfactants

BY RACHEL C. HEALD
Schimmel & Co., Inc.

When the quaternary ammonium salts first came into use as germicides and surfactants, they were often referred to as "invert soaps." Like the fatty acid soaps, they have a long hydrocarbon chain in the molecule which is responsible for their surface-active properties. However, the quaternary ammonium compounds behave in the opposite way from the soaps on ionization. When a soap molecule is dissociated in water, the long chain forms a part of the negative ion or anion. A quaternary ammonium salt ionizes so that the hydrocarbon chain forms a part of the positively charged ion. Since the hydrophobic group of the molecule is in the cation rather than the anion, the salt is an "invert soap."

$RCOONa$	$\longrightarrow$	$[RCOO]^-$	$+ Na^+$
Sodium soap		Active anion	
$R(CH_3)_3NBr$	$\longrightarrow$	$[R(CH_3)_3N]^+$	$+ Br^-$
Quaternary ammonium bromide		Active cation	

(R represents an alkyl group or mixed alkyl groups from C_{12} to C_{18})

The quaternary ammonium germicides are generally regarded as entirely incompatible with fatty acid soaps. When a quaternary is combined with soap in an aqueous solution in roughly equal parts, the two surfactants react to form a wax-like product. This material is not very soluble in water and comes out of solution as a precipitate or suspended particles. If the mixture is very concentrated, it may thicken to a paste. The reaction product is a salt of high molecular weight, formed by the union of the hydrophobic anion of the soap with the hydrophobic cation of the quaternary ammonium compound. Salts of this type are nonpolar and are designated as electro-neutral salts in the patent literature.

Compatible Soap-quaternary Mixtures

Actually, the incompatibility between soaps and quaternary ammonium salts does not extend over the whole range of possible proportions of such mixtures. Aylesworth[1] found that clear solutions result when a soap is mixed with a minor amount of a quaternary ammonium salt (less than 25% of the weight of the soap). The variations in compatibility can be shown by making mixtures of a 4% solution of Hyamine 1622* (quaternary) and a 4% solution of the potassium soap of coconut oil. Both solutions, prepared with distilled water, are clear. When they are combined in equal parts, they give a milky solution. When 80 parts of the Hyamine 1622 solution and 20 parts of the soap solution are mixed, a cloudy liquid results which separates into two layers on standing. But when these proportions are reversed (80 parts soap to 20 parts quaternary) the mixture forms a clear solution.

The maximum amount of quaternary ammonium salt which is compatible with a given soap depends on the molecular weight and solubility of both components of the mixture. The quaternaries of low molecular weight generally are more compatible with soaps than those of high molecular weight. Soap-quaternary mixtures which form clear solutions in distilled water also cause surprisingly little clouding in hard water. The cationic surfactant in the mixture seems to act as a disperser for the insoluble calcium and magnesium soaps which form in hard water.

Aylesworth[1] gives some turbidity readings made on solutions of soap-quaternary mixtures in water of

* Diisobutyl phenoxy ethoxy ethyl dimethyl benzyl ammonium chloride (Rohm & Haas Co.)

100 p.p.m. hardness. The soap used was a potassium coconut soap. The solution of soap alone (0.125%) when tested with a Hellige Turbidimeter gave a reading of 31.** A solution (0.125%) of a mixture of the soap (80%) and lauryl dimethyl benzyl ammonium chloride (20%) gave a reading of only 14. Mixtures of the soap with other quaternary ammonium salts at an 80:20 ratio gave similarly low turbidity values.

Quaternaries in Hard-water Soaps

While these soap-quaternary mixtures do cause some cloudiness in hard water, the precipitate is very finely dispersed. It does not agglomerate like the usual soap curd, so no sticky scum is formed in the wash basin. Most of the commercial cationic surfactants are effective as lime soap dispersers. Coconut soaps require the addition of about 10% of their weight of a quaternary ammonium salt to prevent scum formation in hard water. Soaps containing tallow fatty acids may require up to 20% of quaternary.

As would be expected, the presence of a quaternary ammonium salt in a soap has some effect on its foaming capacity. The soap-quaternary mixtures foam better than the pure soap in hard water at very low concentrations, i.e. when there is insufficient soap to react with all the hardness. However, at higher concentrations, when there is an excess of soap in the water, the quaternary does interfere somewhat with foaming of the soap. In mixed fatty acid soaps this effect can be counteracted by using a higher proportion of coconut fatty acids.

To test the value of a quaternary ammonium salt as an additive in toilet soaps, two batches of the sodium soap of coconut fatty acids were prepared.[1] One batch contained no quaternary while the other contained 7.3% of (3-stearamido propyl) dimethyl ethyl ammonium ethosulfate. Bars of these soaps were used by 21 individuals for routine tub bathing in city water having a hardness of 200 p.p.m. The users judged the soaps primarily on the presence or absence of scum, but also on general performance. All individuals rated the soap containing the quaternary as either good or excellent, while only four rated the control soap in these categories. Eleven rated the control soap as poor. Thus the improvement in the resistance of soap to hard water produced by incorporating a quaternary ammonium salt is readily apparent under conditions of use.

Mixing Cationic Germicides and Anionic Detergents

The quaternary ammonium salts are not very effective as germicides in cleaning solutions based on anionic detergents. They tend to form insoluble reaction products with alkyl sulfates and alkyl aryl sulfonates as well as with soaps. A cationic germicide, when incorporated in a solution of an anionic detergent, loses much of its bactericidal activity and may be entirely inactivated. In turn, it has an adverse effect on the detergency of the anionic compound. The solutions of such mixtures are generally unattractive in appearance because they contain a precipitate or separate into layers.

** Distilled water (blank) gave a reading of 4.

The incompatibility of catonic germicides with anionic detergents can be overcome by combining them with a nonionic solubilizer, as described in the patent of Lambert.[2] Nonionics belonging to the class of alkyl phenol polyglycol ethers are particularly useful in such mixtures as they are good detergents. Since the solubilizer prevents the precipitation of an electro-neutral salt, the mixtures yield concentrated solutions which are clear and fluid. They also remain clear when diluted for use, e.g. for cleaning and sanitizing laundry.

In one example, a solution of 20 parts of a potassium soap and 2 parts of 8-hydroxyquinolinium sulfate in 78 parts of water separated into a top turbid layer and a clear bottom layer. However, a solution of the same active ingredients made up with the addition of 5 parts of a nonionic surfactant remained homogeneous and clear. The nonionic ingredient in this case was the polyethylene oxide derivative of dibutyl phenol.

The incorporation of an alkyl phenol polyglycol ether improves the germicidal activity and detergency of such mixtures, as well as the solubility. The proportion of nonionic surfactant in the mixture must be at least 10% of the combined weight of the ionic surfactants to insure satisfactory germicidal activity.

Anion-cation Products in Hair Preparations

The products formed by the reaction of a cation-active compound with an equivalent amount of an anion-active substance are interesting as cosmetic ingredients and bases. They are oil-soluble materials which are dispersible in hot water. The dispersions have a cream-like consistency and are transparent in thin layers. As the electro-neutral salts are very rapidly adsorbed on the hair from a water base, they are useful in compositions for coloring hair.

The electro-neutral salts may be prepared by mutual precipitation of the anionic and cationic components from a water solution. They are also made by combining a free cationic base (not quaternized) with the acid form of an anionic surfactant, e.g. a fatty acid.[3] A typical anion-cation compound is made by heating together equivalent amounts of stearic acid and dihydroxy diethylene triamine distearylamide. The product is a waxy material.

Dispersions of the electro-neutral salts at a concentration of 2% to 10% are useful as vehicles for hair dyes.[3] Since they are more transparent than emulsified dye creams, they permit the operator to see the development of the color on the hair. Dye preparations containing these products wash out of the hair more readily than the usual dye creams. If the shampoo contains a different anion from the one in the electro-neutral salt, it causes the thick dispersion to liquefy.

The electro-neutral salts are also useful as thickeners for solutions of ammonium thioglycolate. The resulting creams do not feel slippery on the hair like emulsified permanent waving creams.

Color complexes, formed by the reaction of an anionic or cationic dye with a surface-active compound of the opposite type, play an important role in modern hair coloring formulations. The color creme rinses are based on complexes formed by the reaction of an

ionic dye with a cation-active compound. For a discussion of these formulations, the reader should consult the recent article by Goldemberg.[4]

Anion-cation Products in Emulsions

The insoluble electro-neutral salts can be combined with oils and made into emulsions with the aid of an added emulsifier. A patent of Bohme A.-G[5] describes the preparation of stable emulsions for industrial use containing both cationic and anionic surfactants. The cationic ingredient of these emulsions is an alkyl pyridinium salt.

Equimolar portions of this salt and an anionic detergent are reacted in oil solution to form the electro-neutral salt. A small quantity of either the pyridinium salt or the anionic compound is then added and the mixture is emulsified with water. The choice between cationic and anionic emulsifier depends on the use which will be made of the emulsion. In the example below, the cationic ingredient is used as the emulsifying agent.

> Paraffin oil (700 g.) is emulsified in the cold with 140 g. of sodium lauryl sulfate (30% fat content) in a boiler provided with a stirrer. Technical lauryl pyridinium bisulfate (40%) is added thereto until the resulting paste has attained the maximum viscosity. When this point is reached the brine can be separated by heating. The electro-neutral compound, which occludes the paraffin oil, is washed with hot water. It is then converted by further addition of lauryl pyridinium bisulfate into a positively charged emulsion. Because the emulsion is practically free from inorganic salts it is unusually stable.

Directions for preparing a cosmetic cream which contains an anion-cation reaction product are given in a patent of Boehme Fettchemie G.m.b.H.[6] The emulsifying agent in this case is an anionic surfactant, sodium lauryl sulfate.

> Melt together 135 parts by weight of cetyl-myristyl alcohol and 6 parts of glycerol. Sitr into the melt a solution of 2.5 parts of sodium lauryl sulfate in 77 parts of water. Incorporate in the emulsion 10 parts of the salt made from equimolar amounts of dimethyl cyclohexyl dodecyl ammonium chloride and hexadecyl sodium sulfate. Heat the mass with perfume and homogenize. The product is a cream of soft consistency.

The electro-neutral salts are absorbed very rapidly by the skin and function as emollients. Because of their affinity for the skin, they also act as fixatives for the perfume in cosmetic creams. The anion-cation products are useful in formulations containing deodorizing agents or insect repellents, since they bind the active ingredients to the skin.[6]

From the foregoing discussion it is evident that the combination of a cation-active compound and an anion-active compound in the same formulation is not always disastrous. It may result in an improved product, as in the case of hard-water soaps containing a quaternary ammonium salt. Difficulties which arise from the insolubility of the electro-neutral salts often can be overcome by the use of a nonionic solubilizer. The insoluble complexes themselves are now recognized as substances which are beneficial to the skin and useful as cosmetic bases. Since cationic and anionic surfactants are available in a wide range of types and molecular weights, they offer unlimited opportunities for experimenting with new combinations.

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Progress in perfumery materials

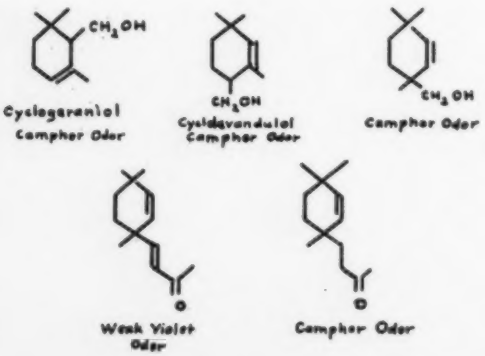
BY PAUL BEDOUKIAN

Continued from March, 1960, pages 51 through 56

Of recent development and becoming increasingly popular are the polyalkylindan compounds which possess powerful and very pleasant odors. The synthesis of a wide range of these compounds is described in the literature (113) (114) and covered by several patents in various countries (115) (116) (117).

Three highly informative articles deal with chemical products having musk odors (118) (119) (120).

Ionone and Irone A number of cyclohexene compounds have been synthesized and their odors noted. This work was carried out in connection with the study of substances possessing the odor of violets and is a notable contribution to our knowledge of odor and its relation to chemical structure. The following substances were found to possess the odors listed (121):



A French patent in 1957 described the condensation of alpha ethylenic alcohols with beta ketonic acids to give alpha ethylenic ketones. Good yields of 2-methyl-2-hepten-6-one, 2,3-dimethyl-2-hepten-6-one, and 2,6-dimethyl-2,6-undecidien-10-one are claimed. These compounds are used in the manufacture of linalool, 3-methyl linalool and nerolidol (122).

A British patent describes the condensation of dehydrolinalool with ethyl acetoacetate to give a mixture of pseudoionones which can then be cyclized to ionones (123). A Swiss patent describes the well-known condensation reaction of dehydrolinalool with diketenes (124).

A novel method of preparing pseudoionone consists of treating such compounds as geranyl halide with alkali and acetone in the presence of nitronic acid. Related compounds such as pseudo-irone and others can also be obtained by starting with the suitable halide (125).

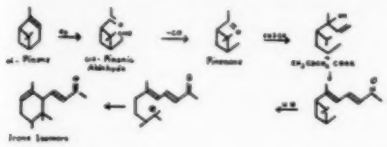
Citral is still employed to a large extent for the manufacture of ionones and methyl ionones. According to a recent study (126) and patent (127), if condensation of citral with butanone is carried out under controlled conditions using suitable catalysts, the resultant product is mainly pseudo-isomethyl ionone which on cyclization yields the highly valued iso-methyl ionone.

Cyclization of various pseudoionones with concentrated sulfuric acid at -60°C. gives almost wholly the alpha isomer, but at higher temperatures the beta isomer is formed increasingly, and at 10°C. practically no alpha ionone is formed. It appears from this study that alpha ionone is the primary cyclization product and it isomerizes to beta ionone at higher temperatures.

A Japanese article discusses the ring-closure of pseudoionone at some length (130).

Some of the reactions reported above have naturally been applied to the preparation of irones and related compounds. Thus, according to two British patents, dehydro-3-methyl linalool was condensed with ethyl acetoacetate to give pseudorione, which on cyclization with phosphoric acid gave alpha irone containing practically no cis-2,6-alpha irone. If the cyclization is carried out with boron fluoride, the resultant product is largely cis-2,6-alpha irone with considerable trans isomer (131) (132). A German patent also deals with this reaction (133).

A novel five-step synthesis of irone reported recently involves the following series of reactions (134):



It is rather involved, and so far there has been no report of a simple and inexpensive synthesis of this valuable compound.

Not only is the synthesis of irone difficult, but it is necessary to have the right isomers in order to approximate the much desired odor of orris root. Some of the difficulties in the analysis of a mixture of the isomers of irone are pointed out in a recent publication (135).

Compounds Having Amber Type Odors. Cyclization of alpha and gamma cyclohomogeraniol gave an oxide which had an amber odor. Cyclohomocitral was found to have a camphor-like odor (136).

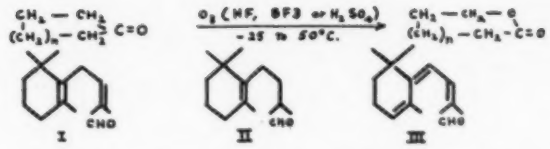
A British patent describes the oxidation of sclareol to give a product having an ambergris odor (137). In a Russian publication, sclareol is oxidized to a lactone which on reduction gave a glycol. High yields of an oxide having an amber-like odor were obtained by dehydrating this glycol (138).

Cyclization of homofarnesylic acid ester gave a lactone which can be used in the synthesis of amber odor compounds (139).

Other publications deal with the cyclization of the ethyl ester of beta hydroxyfarnesyl acetic acid (140) and degradation of manool (141) to give products related to ambreinolide. Since both sclareol and manool are useful in the synthesis of amber odor compounds, the conversion of sclareol to manool is of more than academic interest (142) (143).

Reports on Aromatic Chemicals. There seems to be increased interest in cyclopentanones which have powerful and desirable odors related to jasmine. An excellent review article enumerates the various syntheses of jasmone and dihydrojasmone, as well as the so-called isojasmones and related compounds (144). A new synthesis of dihydrojasmone involves the cyclization of 2,5-dioxo undecane (145). Homoterpenyl methyl ketone has been converted to 3-isopropenyl cyclopentanone (146). It is reported that treatment of delta ethylenic acids with polyphosphoric acid gives mainly cyclopentanones along with cyclohexenones (147). On passing pinonic acid over thorium oxide at elevated temperatures, the cyclobutane ring was found to undergo rupture followed by formation of cyclopentane ring, giving 5-acetyl-3-(1-hydroxymethyl)-cyclopentanone (147a).

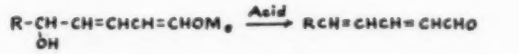
The Villiger synthesis of lactones from cyclic ketones has been applied to a number of cyclopentanones and cyclohexanones, giving the corresponding valerolactones and caprolactones (148). Ozonolysis of alkanones in the presence of various acid catalysts gives esters while cycloalkanones give lactones, according to a recent American patent (149).



A number of recent patents describe the synthesis of higher homologs of cyclocitral. The following three are reported to be useful as perfumery materials, I, II, III, the first two having ionone-like odors (150) (151) (152).

Beta chlorovinyl ketones were converted to alpha beta unsaturated aldehydes by first converting them to their betaoxacetals, reducing them to the beta hydroxyacetals and then obtaining the corresponding unsaturated aldehydes by treatment with acids (153). Another publication describes the formation of beta chlorovinyl ketones by treatment of acid chlorides with acetylene and then converting them to alpha beta unsaturated aldehydes through a similar process described above (154).

It has been found that 2,4-dienals are obtained by treatment of enol vinyl ethers with acids (155).



2-cis, 6-cis nonadienal and its acetal and alcohol, were synthesized through a series of reactions according to a recent American patent (156).

Another patent describes the preparation of aldehydes from primary halides and from mono and disulfonate esters of primary alcohols (157).

Recent developments have indicated that it is commercially feasible to convert 1-alkenes to primary alcohols and one method of this process is covered by several patents (158) (159) (160). The 1-alkene is converted to its aluminate by treatment with isobutyl aluminate and then subjected to oxidation under controlled conditions. A somewhat different process is described in another patent for the same reaction (161).

Allylation of guaiacol gave eugenol, and of anisole gave estragole, as reported in a Russian patent (162). A detailed account of the manufacture of isoeugenol from guaiacol is given in a recent publication (163). Alkylation of eugenol in cinnamon leaf oil or clove oil is described in an Indian patent (164).

Adaptation of a laboratory procedure for industrial scale manufacture of vanillin from guaiacol is described in a recent article (165). A recent patent discloses details for the conversion of vanillin to ethyl vanillin (166).

Phenyl acetaldehyde polymerizes to a crystalline trimer which has been shown to be 2,4-dibenzyl-5-phenyl-6-hydroxy-1,3-dioxane (167). An American patent describes the manufacture of phenylacetaldehyde by treatment of cyclooctatriene with peracetic acid (168).

Hydrogenation of alpha methyl styrene under pressure and in the presence of aluminum and ethyl aluminate gave a product which on oxidation was converted to the alcoholate of hydratropic alcohol (169) (170).

A recent review article describes the preparation of indole and some of its homologs, and their uses in perfumery (171). Another article gives a synthesis of skatole by the cyclization of ortho ethyl formanilide (172).

A Japanese paper gives new synthetic methods of cyclamen aldehyde and related compounds, including 2-methyl-5-isopropyl alphe-methylhydrocinnam-alde hyde. Of the other compounds mentioned, several cyclohexyl derivatives were prepared, of which 2-oxo-3-methyl-4-cyclohexylbutane was found to possess a vetivert type of odor (173). A recent American patent deals with the preparation of 4-tert.-butyl-alpha-hydrocinnamic aldehyde (174).

Heating carbonyl compounds with acetic anhydride in the presence of hydrogen and Raney nickel was reported to give good yields of the acetate of the corresponding alcohol (175).

A large number of derivatives of cyclopentenyl-2-butanone were reported as being useful in perfumery (176).

Myrcene yielded a range of new odoriferous compounds with 1,1-dimethyloctahydronaphthalene skeleton, and these were discussed in a recent article (177).

Among the new aromatic compounds investigated, 6,8-dimethyl-5-noen-2-one is reported to possess a lily-of-the-valley odor (178).

Various condensation products of isophorone and pulegone with alkyl halides have been reported, some of which may prove to have interesting odor qualities (179). Starting from para cymene, 2-methyl-5-isopropylbenzaldehyde and the condensation product with heptaldehyde have been prepared (180). A large number of alkylamino pyridines and alkyl pyridine ethers were prepared and the relation between their structure and odor studied (181).

Interesting Acetylenic Compounds

Acetylenes have been attracting increasing attention because of their interesting odor properties. They can be used in the manufacture of ethylenic compounds.

Two excellent and lengthy articles review the reactions of acetylene compounds and their application to the perfume industry (181a) (181b).

It is curious that hydrogenation of 2-nonynal with palladium catalyst gave the trans isomer of 2-nonenal instead of the expected cis isomer. The semicarbazone of 2-nonynal on similar hydrogenation gave the drivative of cis-2-nonenal. This on treatment with formic acid again gave the trans isomer of 2-nonenal (181c).

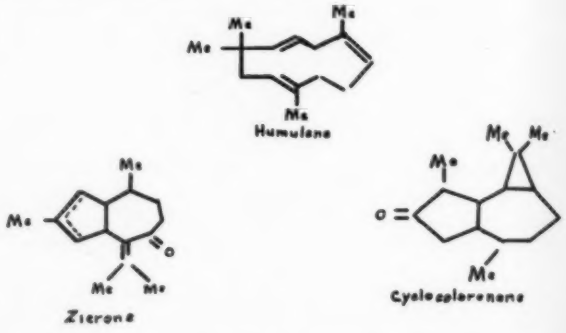
Treatment of monosubstituted acetylenes with orthoesters in the presence of zinc halides, gave acetylenic acetals. (181d) Gamma acetylenic acetals were obtained by treating substituted lithium acetylides with gamma bromo propionaldehyde acetals (181e).

Acetylenic alcohols are obtained by treating alkyl acetylenes with formaldehyde in the presence of copper catalysts. The preparation of 2-octyn-1-ol with this method is reported (181f). It was reported that 1-heptyne readily condenses with chloral in the presence of aluminum chloride (181g). A series of experiments describe the condensation of a range of acetylenes with carbonyl compounds to give acetylenic secondary alcohols (181h). Tertiary acetylenic alcohols are obtained by reacting ketones with acetylenes using suitable catalysts. (181i).

Research in Sesquiterpenes. A great deal of research is being done in this field and as a result, the structure of a large number of sesquiterpenes has been elucidated.

Both sclareol and 13-episclareol have been synthesized recently and studied (182). Various reaction products of sclareol were also investigated by other workers (183).

The steric configuration of acorone and of its stereoisomers was studied (184). Other investigations by the same workers include research on the constitution of ledol (185) (185a) and the ten-membered ring sesquiterpenes, arctiopicrin and germacrane (186 (187). Humulene was given the interesting eleven-membered ring structure (188).



Zierone is shown to be identical with elleryone and its structure proposed as illustrated (189). Cyclocolorenone has been shown to have the azulenic structure shown (190). The structure of alantolactone is being subjected to an intensive investigation (191).

Various sesquiterpenes isolated from the heartwood of several species of trees are being studied (192). A new lactone named costunolide has been isolated from costus oil (193).

A bicyclic lactone has been isolated from the Reformatski reaction of citral with ethyl bromoacetate. The structure of this lactone has been investigated (194). Geranic acid also cyclizes to give a lactone, the structure of which has been elucidated (195).

Reports on Essential Oils

Each year a number of publications report on the production of commonly used oils, the cultivation of new plants as potentially valuable oil-producing sources, and studies on the composition and analysis of many essential oils.

Sabinene hydrate has been shown to be a constituent of American peppermint oil (196) (197). A simple chromatographic method of drop analysis of peppermint oil has been developed, which is applicable in qualitative analyses of other perfume oils (198). The properties of essential oils from mints grown in Soviet Armenia have been reported (199). Another report deals with mint production in Brazil (199a).

According to a recent patent, waxes can be removed from citrus oils by treating such oils with

esterase enzymes under suitable conditions and then freezing and centrifuging the mixture (200). An excellent report has been published on the methods of extraction, analysis, etc. of Florida cold pressed oil (201). A German publication describes the commercial extraction and refining of Orange oil in California (202).

The structure of cadinol obtained from citronella Java was re-investigated (203). It is claimed that ultraviolet spectrophotometry gives results superior to the conventional methods of analysis of lemongrass oil (204).

An interesting report has been published on the cultivation of lavandin and lavender in Languedoc (205). The properties of the oil from lavender hybrids of Yugoslav origin have been studied (206).

The physiochemical properties of various petitgrain oils of Indian origin were published recently (207). Vacuum fractional distillation of Spanish rosemary oil gave a terpeneless oil amounting to one-third of the crude oil (208). The hydrocarbon-free Puerto-Rican bay oil was also studied (209). The composition of Luxembourg cognac oil was published in a German periodical (210).

Costus oil possesses some unusual properties and its composition has recently been reported (211). Another unusual perfumery material is cistus labdanum, and this also is described in a recent publication (212).

Soviet chemists have been studying the composition of vetivert oils and report some new sesquiterpene compounds in this oil (213). Two primary alcohols, namely bicyclovetivenol and tricyclovetivenol, were found in vetivert oil (214). It is claimed that carbonyl compounds in vetivert oil can be conveniently determined potentiometrically (215).

A Japanese patent describes an improved method of obtaining onion oil from onions (216).

Reports on Miscellaneous Oils Numerous reports have been published on essential oils obtained from various new sources. Most of the oils are of little commercial value at present but their study is of interest and some of these oils may eventually become economically important. The composition of artemisia oils obtained from Soviet plants is given in two publications (217) (218). A third report gives the composition of this oil produced in Ireland (219).

Investigations are being continued on the essential oils of the Orthodon species indigenous to East Asia, and the composition of oils of many varieties have been reported (220).

The essential oil of the wood of Vanillosmopsis erythropappa was found to consist almost entirely of levo alpha bisaoblol (221). A large number of constituents of the volatile oil of pseudowintera colorate were recently reported (222). Other publications deal with the examination of carrot seed oil (223), cumin oil (224), hosho leaf oil (225), origanum vulgare (226), dill seed oil (227) and origanum oils of Yugoslav origin (228).

Some newly recognized constituents of pinus sylvestris include myrcene, terpinolene, p-cymene, 1,4-cineole, 1,8-cineole and acetylacetone. Beta carene was found to be absent (229). Eycalyptus caesia and eucalyptus torquata were found to contain large proportions of a new ketone named torquatone which is thought to be (2,4,6-trimethoxy-3,5-dimethyl) benzyl isopropyl ketone (230).

Fennel oil of Yugoslavian origin was found to contain 50-85 per cent trans-anethole and less than 0.3 per cent of the more toxic cisanethole (231). The cis isomer was found to be much more toxic than the trans isomer (232).

The antiseptic action of one hundred perfume oils on growing cultures of six fungi was studied and reported (233).

Books and Review Articles. A number of excellent review articles have served to keep the reader up-to-date on recent developments. Under the heading of "Advances in Sesquiterpene Chemistry," a lengthy article gives an account of much of the recent work done in this field (234). A Russian article discusses the cyclization reactions of the isoprenoid compounds in a thorough article covering forty pages and including nearly three hundred references (235). The Japanese aromatics industry is described briefly in a recent publication (236).

Various methods of preparation of tertiary alcohols, many of which are of value in the perfume industry, have been discussed in great detail (237).

The British Standards Institute has published specifications for a number of essential oils (238).

The abstracted proceedings of the Second International Congress of Essential Oils appeared in a British journal. Many well known authorities participated in this conference and the proceedings were of great interest to the perfume industry (239).

The Fritzsche Library Bulletin continues to give up-to-date abstract headings covering many phases of

The most exhaustive annual publication in our field is still the Schimmel Report, the last issue having 216 pages and covering the year 1958 (241).

The fourth volume of Gildemeister & Hoffmann's recent series on essential oils, edited by Treibs, was published in 1959 (242).

The second volume of the Chemistry of Natural Products deals with mono- and sesqui-terpenes (243).

Two books on perfumery, one a translation of Gatefosse's French volume (244), and the other published in Prague (245), appeared during the past year.

Methods of analysis of essential oils were published by the Syndicat des fabricants et importateurs d'huiles essentielles et produits aromatiques naturels in France (246).

The Essential Oil Association of the United States has published standards for the following essential oils and aromatics: bois de rose Peruvian, carrot seed, galbanum oil, linalyl propionate, parsley seed oil, lovage, myrrh, origanum Spanish, marjoram Spanish, geranyl propionate, methyl heptine carbonate, rhodinol, ambrette seed oil, hydroxycitronellal dimethyl acetal, cyclamen aldehyde, bromstyrol, p-methoxy acetophenone, vetiveryl acetate, oil styrax, geranyl butyrate (247).

The Perfumery and Essential Oil Record is to be congratulated for its magnificent Golden Jubilee publication. This attractive issue contains many interesting articles by leading authorities as well as informa-

tive advertisements of perfumery and essential oil companies (248).

This is the author's sixteenth annual review article on developments in our industry (249).

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The effect of sorbic acid and other preservatives on organism growth in typical nonionic emulsified commercial cosmetics

BY R. DONALD CHARLES AND PHYLLIS J. CARTER
Atlas Powder Company

OF THE MANY factors which determine the resistance of a cosmetic emulsion to organism growth, one has been distinguished by much attention in recent cosmetic and pharmaceutical technical literature—the effect of nonionic surface active agents on emulsion preservation. Most of the work published on investigations of this problem has been based upon studies of preservatives in laboratory culture media containing nonionic surfactants. The failure of many preservatives, especially the phenolic family, to arrest the growth of organisms introduced into this type of media has been attributed to the formation of a complex between preservative and surfactant which impairs the preservative's antimicrobial powers. Extrapolations of the results of this type of component evaluation into the field of cosmetic formulation have led some workers to what we believe are misleading conclusions. In this paper, we are presenting data which illustrate the need to consider the entire cosmetic formulation in dealing with the problem of preservation.

Evidence for some sort of interaction between preservatives and nonionic materials with surface active properties is abundant in the technical literature of the last ten years in the fields of cosmetics and pharmaceuticals. A review of this work, such as that recently compiled by Beckett and Robinson (1), is beyond the scope of this paper; but it would be appropriate to mention in passing some of the more familiar investigators in this field, such as Fisher (2), Gershenfeld (3), Gregg and Zopf (4), Bolle and Mirimanoff (5), Hadgraft (6), Lawrence and Erlandson (7), deNavarre (8), Lord and Husa (9), Barr and Tice (10), Aalto, Firman and Rigler (11), Patel and Kostenbauder (12) and Wedderburn (13).

The mechanism of the interaction between phenolic preservatives and nonionic emulsifiers has been explained by Higuchi and Lach (14) as the formation of a hydrogen bond between the phenolic hydrogen and one of the ether oxygens of the polyoxyethylene chains which are found in many types of nonionic surfactants. Additional support for this explanation has been contributed by the work of Hall and deNavarre (15).

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The manner in which nonionic surfactant micelles could be solvated by hydrogen bonding and the way in which a phenolic molecule could be attracted to the surfactant micelle and held by a combination of hydrogen bonding and van der Waals forces has been discussed by Beckett and Robinson (1).

Most of these studies have been concentrated on the detection of preservative-surfactant interaction in systems less complex than the cosmetic emulsions in which these ingredients are used. The demonstration of the effects of preservative complexing in these systems has been made on the basis of reduced activity of the preservative toward organisms introduced into a laboratory culture medium containing nonionic surfactants, or the proof of interaction has been based on physical measurements of dilute two and three component systems with water, such as dialysis studies (12) or turbidity determinations (16). Very little has appeared in the literature, however, in the way of documented correlations between the behavior of a preservative compound in a nonionic surfactant-nutrient broth combination and the same preservative's behavior in a cosmetic emulsion.

Lacking such a conversion factor, the other possibility of relating component testing to practice is the testing of the complete preserved formulation for resistance to organism growth. One of the few published examples of this type of work has been presented by Rdzols, Grundy, Kirchmeyer and Sylvester (17), who tested a group of pharmaceutical products by introducing a suspension of garden soil with its normal flora supplemented by the addition of the spores of nine molds.

In the course of a study to determine the effectiveness of sorbic acid as a preservative for cosmetic emulsions based on nonionic surfactant systems, conducted for the Atlas Powder Company by Tice and Barr of the Philadelphia College of Pharmacy and Science, we had occasion to have three typical cosmetic formulas tested for their resistance to organism growth.

These formulas were o/w emulsions containing nonionic surfactant systems and were first prepared with no preservative, then preserved with a widely used methyl *para*hydroxybenzoate/propyl *para*hydroxybenzoate combination and finally protected against organism growth with sorbic acid.

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Portions of the three modifications of each cosmetic preparation were then contaminated individually with six separate microorganisms, and the samples observed during a storage period of three months at room temperature.

Some of the results of this portion of the study alluded to in a paper presented by Tice (18) before this Society on December 10, 1957, suggested to us that cosmetic emulsions containing nonionic surfactants and preserved with *para*hydroxybenzoate esters would resist organism growth with more success than was indicated by the testing of the preservative in nutrient culture media containing nonionic surfactants and that sorbic acid had value as a preservative for cosmetic emulsions containing nonionic surface-active agents.

In order to see if the discrepancy between observed preservative behavior and that predicted (1) for preservatives in nonionic emulsions was peculiar to our formulations or could be observed in nonionic cosmetic emulsions in general, we decided to extend this study to typical commercial cosmetic emulsions based on nonionic surfactants. Through the courtesy and cooperation of four nationally known cosmetic houses, we were able to obtain for testing two series of samples of commercial nonionic cosmetic emulsions. The two series of samples in each case differed only in the nature and quantity of the preservative included in each formulation. The first series of cosmetic samples contained the preservative system which is being used currently by the manufacturer to prevent organism growth in the cosmetics, while the second series contained sorbic acid in the place of the customary preservative.

The suppliers of these samples also provided information on the nature of the product and a partial list of ingredients which included: (a) the type and concentration of nonionic emulsifier system; (b) the type and concentration of preservative; (c) the type and concentration of other major ingredients in the formulations which might exert preservative action. Because these formulations are commercial items, a more complete list of ingredients and their relative concentrations was not revealed by the manufacturers. Knowing only the nature and concentration of the nonionic surfactant and preservative systems does not permit us to speculate about the relative importance of other factors which influence a cosmetic emulsion's resistance to organism growth, such as the contribution of dyes (19, 20) and perfume oils (21) to the preservative system or the various combinations of nonsurface-active and nonpreservative cosmetic ingredients which could make one formulation a more nutrient medium than another (18). The acidity or alkalinity of the cosmetic emulsion also contributes to the total picture of preservation, but our attention in this investigation was focused on the interaction between preservative and nonionic emulsifier in the finished cosmetic formulation. If the interaction between preservative and nonionic emulsifier is the controlling factor in the preservation of these nonionic cosmetic emulsions, and if this interaction occurred to the extent indicated by component testing of nonionic surfactant and preservative in microorganism culture medium (1, 8), these commercial cosmetics would

be expected to support organism growth with preservation being the exception rather than the rule. If, on the other hand, the preservative-nonionic surfactant interaction is not so great as indicated by the evaluation of less complex systems (as suggested by the data in Table 1) and especially if this interaction is modified by the emulsion system in which the preservative and surfactant are used, the cosmetic emulsions should be resistant to organism growth in general, with no general pattern discernible from the limited information available on the compositions of the commercial cosmetic emulsions tested.

In the hope of observing one of the above specified patterns of organism growth, portions of the two types of each commercial nonionic cosmetic formulation which differed only in the preservative employed were contaminated individually with each of nine different microorganisms, and the samples observed for organism growth during three months' storage at room temperature.

The observations of these samples, collected in Tables 2, 3, 4 and 5, reinforce the results of the previous experiment in that they suggest further that the activity of a phenolic preservative in a nonionic cosmetic emulsion is not always so inhibited as component testing suggests and that sorbic acid seems to offer adequate preservation in emulsion systems where the *para*hydroxybenzoate esters fail. In addition, these results indicate that each cosmetic emulsion should be considered individually and tested as a whole when preservation problems arise.

1. Preparation of Three Typical Nonionic Cosmetic Formulations

A. O/W Cold Cream

Mineral oil, beeswax and surfactants were combined and heated to 70°C. The water was heated to 72°C. and added slowly with propeller agitation to the mixture. The emulsion was then allowed to cool to room temperature, with an anchor-type agitator being substituted for the propeller as the cream reached the "set" point. In the preparation of the preserved samples, the preservatives were added to the water prior to its being heated to 72°C. and added to the mixture of beeswax, mineral oil and surfactants.

B. O/W Face Cream

Stearic acid, isopropyl myristate-palmitate and surfactants were combined and heated to 60°C. The water and 70 per cent sorbitol solution were combined and heated to 62°C., after which this mixture was added to the surfactant-fatty acid-fatty ester combination in the manner described for the o/w cold cream. Preservatives were incorporated into the face cream as described with the preparation of the cold cream.

Table 1—The Resistance of Three Typical Nonionic Cosmetic Emulsions to Organism Growth During a Period of Three Months

Type of Cosmetic Product	Cold Cream, o/w	Face Cream, o/w	Baby Lotion o/w
Emulsion Type Ingredients (% w/w)			
Mineral oil	50.0	..	35.0
Beeswax	7.0	..	..
Lanolin	..	..	1.0
Cetyl alcohol	..	..	1.0
Stearic acid (triple pressed)	..	15.0	..
Isopropyl myristate-palmitate	..	1.0	..
Tween® 40 (P.O.E. sorbitan monopalmitate)	2.0	..	..
Tween® 60 (P.O.E. sorbitan monostearate)	..	1.5	..
Tween® 80 (P.O.E. sorbitan monooleate)	..	..	4.9
Arlacel® 60 (sorbitan monostearate)	..	2.0	..
Span® 80 (sorbitan monooleate)	..	..	2.1
G-1726 (P.O.E. sorbitol beeswax derivative)	8.0	..	..
Sorbo® (70% sorbitol solution)	..	3.0	..
Water	33.0	77.5	56.0
Preservatives			
Methyl parahydroxybenzoate	.. 0.18 ..	.. 0.18 ..	.. 0.18 ..
Propyl parahydroxybenzoate	.. 0.05 ..	.. 0.05 ..	.. 0.05 ..
Sorbic acid	.. .. 0.2	.. .. 0.2	.. .. 0.2
Growth of test organisms in samples during a period of three months			
Control sample	− − −	− − −	− − −
Exposed to air	+ − −	+ − −	+ − −
Bacterium, *Pseudomonas aeruginosa*	+ − −	+ − −	+ − −
Molds			
Aspergillus niger	+ + −	+ − −	+ − −
Cladosporium carpophilum	+ − −	+ − −	+ − −
Mucor plumbeus	+ − −	+ − −	+ − −
Penicillium chrysogenum	+ − −	+ − −	+ − −
Rhizopus nigricans	+ − −	+ − −	+ − −

o/w—oil in water. % w/w—weight per cent. P.O.E.—polyoxyethylene. I.U.—international units. ®—registered trademark, Atlas Powder Company.

Organism growth: +, growth observed; −, no growth apparent.

C. O/W Baby Lotion

Mineral oil, lanolin, cetyl alcohol and surfactants were combined, heated and combined with water by the method described for the preparation of the face cream, except that the emulsion was allowed to cool to room temperature with propeller agitation.

2. Resistance of Three Typical Nonionic Cosmetic Emulsions to Organism Growth

A. Organisms Employed in the Investigation (23)

Bacterium—*Pseudomonoas aeruginosa.*

Molds—*Aspergillus niger, Cladosporium carpophilum, Mucor plumbeus, Penicillium chrysogenum, and Rhizopus nigricans.*

B. Contamination of the Samples

One-hundred gram quantities of each of the three variations of each preparation containing respectively no preservative, a methyl and propyl *para*hydroxybenzoate combination, and sorbic acid were placed in four-ounce bottles and contaminated by surface inoculation and by stabbing with cultures obtained from the following sources:

(1) *Molds*—a seventy-two-hour Fluid Sabouraud Medium suspension or a seventy-two-hour Sabouraud Agar growth

(2) *Bacterium*—a twenty-four-hour nutrient broth suspension or a twenty-four-hour nutrient agar growth.

All inoculations were made in duplicate. One set of samples was not inoculated but exposed to the air for one hour daily, while another set of samples was used as uninoculated controls.

C. Observation of the Samples

The samples were observed during a storage period of three months at room temperature for visible growth of microorganisms, production of odors and any physical change which would make the cosmetic formulation unusable or unsatisfactory. The observation data presented in Table 1 are based on samples receiving a broth culture inoculum because this source of intentional contamination produced, for the most part, quicker and more luxuriant organism growth than did the inoculum of slant cultures.

3. Resistance of Commercial Cosmetic Emulsions to Organism Growth

A. Organisms Employed in the Investigation (23)

Bacterium—*Pseudomonas aeruginosa.*

Molds—*Alternaria tenuis, Aspergillus fumigatus, Aspergillus niger, Cladosporium herbarum, Mucor plumbeus, Penicillium chrysogenum, Penicillium notatum and Rhizopus nigricans.*

B. Contamination of the Samples

This was conducted generally as before with two exceptions. First, the organisms were introduced to the cosmetic emulsions in an aqueous suspension rather than in a portion of their culture medium. This method of contamination was used to force the organisms to attack the cosmetic preparation immediately in order to multiply rather than allowing them to grow initially. One-tenth of one milliliter of an aqueous suspension of each type (about 1,500,000 microorganisms) of contaminant was introduced into individual four fluid ounce portions of both preservative variations of the commercial cosmetics. The second exception was the elimination of the samples which were subjected to daily airborne contamination.

C. Observation of the Samples

The inoculated samples as well as uninoculated controls were stored at room temperature for a period of three months and observed periodically as before for evidences of microorganism growth and detrimental changes in the emulsions.

In the first testing sequence involving three typical nonionic cosmetic emulsions (Table 1), one of the 21 contaminated *para*hydroxybenzoate preserved emulsions supported organism growth during the three-month observation period, none of the 21 contaminated sorbic acid preserved samples supported organism growth, and all of the 21 contaminated emulsions which had no preservative supported organism growth. These results indicate, especially in the absence of materials which would act as auxiliary preservatives or preservative synergists in any of the three formulations, that the extent to which preservatives have been shown to be inactivated by nonionic surfactants in organism culture media or dilute aqueous solutions or dispersions does not always parallel the preservative's behavior in a cosmetic emulsion based on a nonionic surfactant system.

Discussion of the results of the determination of resistance to organism growth of the 18 commercial nonionic cosmetic emulsions is restricted by the scope of this paper and the limited information made available to us to consideration of whether the nonionic surfactants used in all of these preparations inhibited the present preservative systems, composed mainly of *para*hydroxybenzoate esters, and the alternate sorbic acid preservative system to the extent that the finished cosmetic products supported organism growth. Ingredients in each preparation other than preservative and surfactant which could be capable of making a major contribution to the preservative system were noted, but further contributions from other possible components such as dyes, perfume oils, astringent salts or mild bases could not be assessed on the basis of our limited knowledge of the formulation even though it has been suggested (18, 23) that the degree of preservation in a cosmetic emulsion is a problem which embraces the entire formulation. With this latter point in mind, an examination of the data presented in Tables 2, 3, 4 and 5 showing the differing behavior of two preparations which appear from our restricted knowledge of their compositions to be identical can be attributed to differences in the unidentified components of each emulsion and the final differences which they in turn cause in both finished preparations.

The results of testing six lotions with two different preservative systems are presented in Table 2. Of the six lotions tested, Lotion 2 when preserved with a methyl *para*hydroxybenzoate/dehydroacetic acid combination, supported a heavy growth of one of the nine test organisms. None of the sorbic acid preserved samples supported perceptible organism growth. Lotion 1, which appears to be equivalent to Lotion 2

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Table 2–The Resistance of Six O/W Commercial Cosmetic Lotions to Organism Growth during a Period of Three Months*

	Lotion 1		Lotion 2		Lotion 3		Lotion 4		Lotion 5		Lotion 6	
Preservatives												
Methyl parahydroxybenzoate + dihydroacetic acid	0.3	..	0.3	..	0.3	..	..	..	..	..	..	..
Methyl parahydroxybenzoate	..	..	..	..	..	..	0.2	..	0.2	..	0.15	..
Propyl parahydroxybenzoate												
Sorbic acid	..	0.2	..	0.2	..	0.2	..	0.2	..	0.2	..	0.2
Possible auxiliary preservatives												
Ethanol	5.0		5.0		5.0		..		..		..	
Propylene glycol	5.0		5.0		5.0		..		..		..	
5000 I.U. estrogen/ounce*	..		..		..		..		*		..	
Control	–	–	++	–	–	–	–	–	–	–	–	–
Bacterium, *pseudomonas aeruginosa*	–	–	–	–	–	–	–	–	–	–	–	–
Molds												
Alternaria tenuis	–	–	++	–	–	–	–	–	–	–	–	–
Aspergillus fumigatus	–	–	–	–	–	–	–	–	–	–	–	–
Aspergillus niger	–	–	–	–	–	–	–	–	–	–	–	–
Cladosporium herbarum	–	–	–	–	–	–	–	–	–	–	–	–
Mucor plumbeus	–	–	–	–	–	–	–	–	–	–	–	–
Penicillium chrysogenum	–	–	–	–	–	–	–	–	–	–	–	–
Penicillium notatum	–	–	–	–	–	–	–	–	–	–	–	–
Rhizopus nigricans	–	–	–	–	–	–	–	–	–	–	–	–

Nonionic emulsifier system:

Lotion 1–Polyoxyethylene sorbitol lanolin derivative/polyoxyethylene stearyl alcohol–5% w/w.
Lotion 2–Polyoxyethylene sorbitol lanolin derivative/polyoxyethylene stearyl alcohol–5% w/w.
Lotion 3–Polyoxyethylene sorbitol lanolin derivative/polyoxyethylene stearyl alcohol–5% w/w.
Lotion 4–Sorbitan monoester of fatty acid/polyoxyethylene sorbitan monoester of fatty acid–5.5% w/w.
Lotion 5–Sorbitan monoester of fatty acid/polyoxyethylene sorbitan monoester of fatty acid–5% w/w.
Lotion 6–Polyethylene glycol, mol. wt. 1300 to 1600/glyceryl monostearates–8% w/w.

* See Table 1 for guide to abbreviations and symbols.
++, heavy growth observed; +, slight to moderate growth observed; –, no growth apparent.

Table 3–The Resistance of Four O/W Commercial Cosmetic Lotions to Organism Growth During a Period of Three Months*

	Suntan Lotion		Hand Lotion		Foundation Lotion 1		Foundation Lotion 2	
Preservatives								
*Para*hydroxybenzoate esters	0.3	..	0.3	..	0.3	..	0.3	..
Sorbic acid	..	0.2	..	0.2	..	0.2	..	0.2
Possible auxiliary preservative, salicylates –assumed less than	5%		..		..		..	
Control	–	–	–	–	–	–	–	–
Bacterium, *Pseudomonas aeruginosa*	–	–	–	–	–	–	–	–
Molds								
Alternaria tenuis	–	–	+	–	–	–	–	–
Aspergillus fumigatus	–	–	–	–	–	–	–	–
Aspergillus niger.	–	–	–	–	–	–	–	–
Cladosporium herbarum	–	–	–	–	–	–	–	–
Mucor plumbeus	–	–	–	–	–	–	–	–
Penicillium chrysogenum	–	–	–	–	–	–	–	–
Penicillium notatum	–	–	–	–	–	–	–	–
Rhizopus nigricans	–	–	+	–	–	–	–	–

Nonionic emulsifier system:

Suntan lotion–polyoxyalcohol ethers–5% w/w.
Hand lotion–Sorbitan monoester of fatty acid/P.O.E. sorbitan monoester of fatty acid–10% w/w.
Foundation lotion 1–Ethylene oxide condensates of fatty alcohols–10% w/w.
Foundation lotion 2–Sorbitan monoester of fatty acid/P.O.E. sorbitan monoester of fatty acid–10% w/w.

* See Table 1 for guide to abbreviations and symbols.
++, heavy growth observed; +, slight to moderate growth observed; –, no growth apparent.

from the information available to us, supported no growth with either preservative system, suggesting that Lotions 1 and 2 may not be identical after all. The nature of the heavy organism growth observed in the uninoculated, methyl *para*hydroxybenzoate/dehydroacetic acid preserved control for Lotion 2 was not identified.

To commercial foundation lotions, a suntan lotion and a hand lotion, formed the subjects for the testing summarized in Table 3. The concentration of salicylate in the suntan emulsion was not revealed to us, but it was assumed to be less than five per cent on the basis of the tested active ingredient contents of many currently marketed preparations of a similar nature. One of these lotions, the hand lotion, supported a slight to moderate growth of two of the nine test organisms with a *para*hydroxybenzoate ester preservative system, while no contaminant growth was observed in any of the preparations containing sorbic acid.

Table 4 contains the three-month results of the testing of four cleansing lotions for their resistance

Table 4—The Resistance of Four O/W Commercial Cosmetic Cleansing Lotions to Organism Growth During a period of Three Months*

	Cleansing Lotion 1		Cleansing Lotion 2		Cleansing Lotion 3		Cleansing Lotion 4	
Preservatives								
Methyl *para*hydroxybenzoate	0.3	..	..	..	..	..	..	..
Propyl *para*hydroxybenzoate								
*Para*hydroxybenzoate esters	..	..	0.3	..	0.2	..	..	..
Quaternary ammonium salt	..	..	..	..	1.25	..	1.25	..
Sorbic acid	..	0.2	..	0.2	..	0.2	..	0.2
Possible auxiliary preservative, propylene glycol	5.0		..		..		..	
Control	–	–	–	–	–	–	–	–
Bacterium, *Pseudomonas aeruginosa*	–	–	–	–	–	–	–	–
Molds								
Alternaria tenuis	–	–	–	–	–	–	++	–
Aspergillus fumigatus	–	–	–	–	–	–	–	–
Aspergillus niger	–	–	–	–	++	–	++	+
Cladosporium herbarum	–	–	–	–	–	–	–	–
Mucor plumbeus	–	–	–	–	–	–	++	–
Penicillium chrysogenum	–	–	–	–	–	–	–	–
Penicillium notatum	–	–	–	–	–	–	++	–
Rhizopus nigricans	–	–	–	–	–	–	–	–

Nonionic emulsifier system:
Cleansing lotion 1—Sorbitan monoester of fatty acid/ethoxylated fatty acids, glyceride ester, and fatty alcohols —11% w/w.
Cleansing lotion 2—Ethylene oxide condensates of fatty alcohols—6% w/w.
Cleansing lotion 3—Sorbitan monooleate/polyoxyethylene sorbitan monooleate—8% w/w.
Cleansing lotion 4—Sorbitan monooleate/polyoxyethylene sorbitan monooleate—8% w/w.

* See Table 1 for guide to abbreviations and symbols.
++, heavy growth observed; +, slight to moderate growth observed; –, no growth apparent.

Table 5—The Resistance of Two O/W Commercial Vanishing Creams and Two O/W Commercial Hair Grooming Preparations to Organism Growth During a Period of Three Months

	Vanishing Cream 1		Vanishing Cream 2		Hair Groom 1		Hair Groom 2	
Preservatives								
*Para*hydroxybenzoate esters	0.3	...	0.3	...	0.3	...	0.3	...
Sorbic acid	...	0.2	...	0.2	...	0.2	...	0.2
Control	...	...	...	...	...	...	...	...
Bacterium, *Pseudomonas aeruginosa*	...	...	...	...	...	...	...	...
Molds								
Alternaria tenuis	–	–	–	–	++	–	+	–
Aspergillus fumigatus	–	–	–	–	–	–	–	–
Aspergillus niger	–	–	–	–	++	–	–	–
Cladosporium herbarum	–	–	–	–	–	–	–	–
Mucor plumbeus	–	–	–	–	–	–	+	–
Penicillium chrysogenum	–	–	–	–	++	–	–	–
Penicillium notatum	–	–	–	–	–	–	–	–
Rhizopus nigricans	–	–	–	–	++	–	+	–

Nonionic emulsifier system: Vanishing cream 1, polyethylene glycol fatty acid derivatives, 10% w/w. Vanishing cream 2, polyethylene glycol fatty acid derivatives, 10% w/w. Hair groom 1, polyoxyethylated nonyl phenol, fatty acid, and alkylolamide, 6% w/w. Hair groom 2, polyoxyethylated nonyl phenol, fatty acid, and alkylolamide, 6% w/w.

to organism growth. One of the four emulsions supported slight growth with one of the nine test organisms in a sorbic acid preservative system, and two of the lotions supported heavy growths of several of the test organisms when the current preservative system was used. Cleansing Lotion 3 supported a heavy growth of one of the nine test organisms when protected by a quaternary ammonium salt/*para*hydroxybenzoate ester combination. Cleansing Lotion 4 supported heavy growths of four of the nine test organisms with a preservative system supplied by a quaternary ammonium salt, and supported slight to moderate growth of *Aspergillus niger* in the presence of sorbic acid. Although Cleansing Lotions 3 and 4 appear to be identical from the information which is available, the conflicting observations of the apparent improvement protection afforded by the addition of *para*hydroxybenzoate esters to the quaternary ammonium salt and the effectiveness of sorbic acid when substituted for the quaternary ammonium salt/phenolic ester combination but not for the quaternary salt alone could best be resolved by a comparison of the complete formulations of both preparations.

The results of testing two vanishing creams and two hair grooming preparations are presented in Table 5 and depict a situation similar to the one discussed with the cleansing creams. Both vanishing creams appeared to be of the same composition from our limited knowledge; and since neither cream supported organism growth with a *para*hydroxybenzoate system or with a sorbic acid preservative system, no difference in composition was suggested by differing resistances to organism growth. The hair grooming preparations also appear to be the same from the composition information available; and although neither preparation supported organism growth when preserved with sorbic acid, Hair Groom 1 with a *para*hydroxybenzoate ester system supported heavy growths of *Alternaria tenuis, Aspergillus niger, Penicillium chrysogenum* and *Rhizopus nigricans,* while Hair Groom 2 with *para*hydroxybenzoate preservative supported only slight to moderate growths of *Alternaria tenuis, Mucor plumbeus* and *Rhizopus nigricans.* Here, again, a comparison of complete formulations is needed to explain observations which do not coincide when the hair grooming preparations are assumed to be identical on the basis of similarities of type and concentration of emulsifiers and preservatives.

This short study suggests that:

1. The results of an organism inhibition test of a preservative in a laboratory culture medium containing a nonionic surfactant are not always typical of the behavior of that preservative in a finished cosmetic emulsion which includes the preservative and surfactant used in the test. Since little in the way of proven correlation exists between the results of such component testing and preservative behavior in a particular finished product is best determined at the present time by testing the finished product itself for its resistance to organism growth.

2. Combinations of *para*hydroxybenzoate esters and *para*hydroxybenzoate esters with dehydroacetic acid, used to preserve many cosmetic products, will probably provide adequate protection for cosmetic emulsions even in the presence of nonionic surface active agents. Where these preservatives fail, sorbic acid appears to provide adequate protection.

Summary

Three typical cosmetic emulsions based on nonionic surfactants were prepared and preserved first with a mixture of methyl and propyl *para*hydroxybenzoates and then with sorbic acid. Testing of these preparations with six different microorganisms for a period of three months indicated that the phenolic preservatives were more effective in finished preparation than would be expected in light of some studies based on component testing which have appeared in recent technical literature. Where the *para*hydroxybenzoate esters failed in preserving these samples, sorbic acid appeared to provide adequate protection.

Eighteen commercial cosmetic emulsions based on nonionic surfactant systems were obtained from commercial sources in two almost identical groups: one group contained the preservative currently used by the manufacturer to protect it, while the other group used sorbic acid as its preservative. Both modifications of these commercial cosmetics were tested with nine microorganisms for their resistance to organism growth during a period of three months. Results of this evaluation reinforced the findings of the previous study in that the *para*hydroxybenzoate esters were more effective in finished formulations than had been predicted from their performance in component testing and that sorbic acid appeared to afford adequate protection in most cases where the phenolic esters failed.

Both sections of this study support the contention that the preservation of nonionic cosmetic emulsions depends upon more factors than interaction between preservative and nonionic surfactant and that the only consistently reliable way to determine the resistance of a cosmetic emulsion to organism growth is to test the finished product itself.

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Steroidal sapogenins and derived steroid hormones

Dr. Monroe E. Wall, Plant Steroid Investigations
Eastern Regional Research Laboratory

The dramatic announcement of the anti-arthritic action of cortisone by Hench, Kendall and Collaborators in 1949 (1) was responsible for an enormous increase in steroid research. As a result steroid hormones are today responsible for sales in excess of 120 million dollars. The major factor in these figures are the anti-inflammatory corticosteroids accounting for some 80% of total sales. However there is currently increasing interest in a wide variety of androgenic, progestational and arabolic agents which may eventually equal the sales of the corticosteroids.

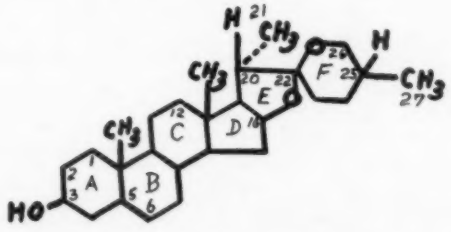
Practically any steroid hormone can be and is being produced from steroidal sapogenins. In this article will be presented some of the salient features of the chemistry and technology of this group of steroids which constitute the most important source of basic chemicals for the production of steroid hormones.

Steroidal Sapogenins

Basic Structure

Steroidal sapogenins occur in nature as plant glycosides which have the property of forming a soapy lather in water and hence are known as saponins. The steroidal saponins hemolyze blood, and are also fish poisons. The saponins are not toxic to humans when ingested orally, probably because of nonabsorption from the intestines. The steroidal saponin, digitonin, from digitalis leaf is a widely used quantitative precipitating agent for sterols such as cholesterol.

Although the occurence of saponins along with cardiac glycosides in digitalis was recognized as early as 1875, the basic structure of the steroidal aglycones derived from the saponins by acidic hydrolysis was not determined until the period 1939-1943. At this time the classic researches of Russell Marker and his students (2) resulted in the complete elucidation of the basic structure shown below in Chart I.



Typical Structure of Steroidal Sapogenins

Marker converted the delta-5-unsaturated sapogenin, diosgenin to cholesterol thus establishing the fact that the sapogenins were C_{27} steroids with rings ABCD identical to those found in all common steroids. The characteristic spiroketal structure of rings E and F was also established by Marker and his co-workers (2). In the formulation shown above it will be noted that the sapogenin sidechain consists of a five and a six-membered ring joined by a spiro carbon atom at C_{22}. Another way of looking at the spiroketal structure is to think of the C_{22} carbon atom as a potential carbonyl group which has formed a full ketal as a result of reaction with the C_{16} and C_{20} hydroxyl groups. There are three asymmetric carbon atoms in rings E and F at C_{20}, C_{22} and C_{25} and hence 8 different isomers are possible. The stereochemistry of the spiroketal sidechain has in recent years received much attention and has been largely elucidated by

researches conducted by R. K. Callow and co-workers at the National Medical Institute, England; E. Mosettig and co-workers at the National Institute of Health, Bethesda, Md., and at the author's laboratory. Details of these investigations are excellently summarized in the recent book by the Fiesers (3), pp. 810-846.

Occurrence of Steroidal Sapogenins in Nature

Although steroidal sapogenins were first discovered in Digitalis leaves, this plant, which is a dicot belonging to the Scrophulariaceae family, is an exception to the general rule. Studies by Marker (2) and more recently extensive surveys at the Eastern Regional Research Laboratory (4) have shown that steroidal sapogenins occur in monocots found in a narrow segment of the Plant Kingdom. As shown in Chart II below, the principal Plant Families are the Libaceae, Amaryllidaceae, and Dioscoreaceae of which Yucca, Agave, and Dioscorea are the important species. In the case of Yucca and Agave, the principal source of sapogenins from a commercial viewpoint are the leaves. However unpublished data from the Eastern Regional Research Laboratory indicates that relatively high concentrations are found in flowers and seeds. In the case of Dioscorea, the tubers are the sole source of the Sapogenin.

Order		
Liliales		
Family		
Liliaceae	Amaryllidaaceae	Dioscoreaceae
Liliaceae	Amaryllidaceae	Dioscoreaceae
Species	Species	Species
Agapanthus	Agave	Dioscorea
Chlorogalum	Manfreda	
Nolina		
Smilax		
Yucca		
Trillium		

Chart II Botanical Relationships of Sapogenaceous Plants.

Sapogenin Types

A wide variety of sapogenins have been found in nature. These steroids may differ in degree of hydroxylation, presence or absence of a 12-carbonyl group, isomerism or the presence of a delta^5 moiety at the Ring A/B juncture, and finally in isomerism at C_{25}. Chart III lists a number of sapogenins with established structure and also gives a typical plant source for each sapogenin. The compilations listed in reference 4 should be consulted for further details. Although a large number of sapogenins are known, only those which are monohydroxylated in ring A have any utility at the present time. The following sapogenins have actual or potential utility; diosgenin, hecogenin, tigogenin, gentrogenin, smilagenin, and sarsasapogenin.

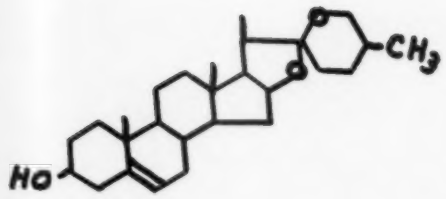
Diosgenin

Occurrence

Because of both its chemical structure and availability, diosgenin is *the* steroidal sapogenin of commerce. Its sources, the tubers of various Dioscorea species are widespread in nature (5). In the Western hemisphere, diosgenin containing species are found as far north as Pennsylvania and continue south through Mexico and Central America and are found even in Northern Chile. Vast commercial areas occur in the Vera Cruz Province of Mexico and also in the

Chart III
Steroidal Sapogenins and Plant Sources

Name	A/B Fusion	Isomerism at C25	Substituents	Typical Plant Source
Sarsasapogenin	β	neo	3β-OH	Y. Baccata
Smilogenin	β	iso	3β-OH	A. lecheguilla
Markogenin	β	neo	2β, 3β-(OH)$_2$	Y. schidigera
Samogenin	β	iso	2β, 3β-(OH)$_2$	Y. carnerosana
Willagenin	β	neo	3β-OH, 12 CO	Y. filifera?
Mexogenin	β	iso	2β, 3β-(OH)$_2$, 12 CO	Y. schottii
Neotigogenin	α	neo	3β-OH	A. sisalana
Tigogenin	α	iso	3β-OH	Y. peninsularis
Gitogenin	α	iso	2α, 3β-(OH)$_2$	A. schottii
Sisalagenin	α	neo	3β-OH, 12 CO	A. sisalana
Hecogenin	α	iso	3β-OH, 12 CO	A. sisalana
Manogenin	α	iso	2α, 3β-(OH)$_2$, 12 CO	A. nelsonii
delta^9-Hecogenin	α	iso	3β-OH, $\Delta^{9(11)}$, 12 CO	A. nelsonii
Chlorogenin	α	iso	3β, 6α-(OH)$_2$	Chlorogalum pomeridianum
Digitogenin	α	iso	2α, 3β-(OH)$_3$	Digitalis purpurea
Yamogenin	Δ^5	neo	3β-OH	D. bartlettii
Diosgenin	Δ^5	iso	3β-OH	D. composita
Yuccagenin	Δ^5	iso	2α, 3β-(OH)$_2$	Y. filamentosa
Kammogenin	Δ^5	iso	2α, 3β-(OH)$_2$, 12 CO	Y. filamentosa
Gentrogenin	Δ^5	iso	3β-(OH), 12 CO	D. spiculiflora
Correllogenin	Δ^5	neo	3β-(OH), 12 CO	D. spiculiflora



Oaxca, and Chiapas provinces of Mexico and in Guatemala. There are some diosgenin containing Dioscorea found in India and in South Africa. Not all Dioscorea contains diosgenin. Some are devoid of sapogenins; others contain yamogeni, a C_{25} isomer of diosgenin and other more hydroxylated steroids; whereas others contain an alkaloid called dioscorine. Some typical tubers are shown in figure 1. The best species in terms of diosgenin content are D. composita and D.

Figure 1. Some typical Dioscorea Tubers.

floribunda which average 4-5% on a dry basis. Individual collections often run higher.

Preparation

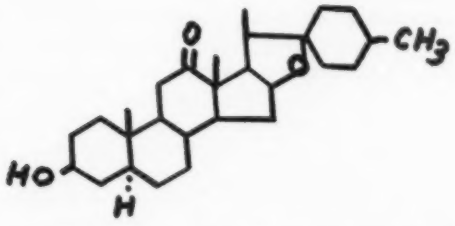
Dioscorea tubers are collected in areas where they grow wild in abundance. Of necessity hand labor is involved but is feasible because of the low wage scales prevailing for this type of work. Under the best circumstances the tubers are brought to central collection points and are there ground and usually allowed to ferment prior to drying. It is essential that the tubers be ground prior to drying as enzymes are released which materially raise the yield of diosgenin. The dried, ground product is then shipped from the Mexican collection areas, such as Vera Cruz province, to Mexico City where are located practically all the important producers of diosgenin.

Diosgenin may be obtained from the ground crude, root in several ways. The ground product may be extracted with hot water or alcohol to remove the glycosidal saponins. Hydrolysis with either hydrochloric or sulfuric acids then gives the crude sapogenin. Under atmospheric pressure it is necessary to use at least 1.5N acid. If one operates under pressure the quantity of acid required is markedly reduced to 0.5N. The crude sapogenin thus obtained is insoluble and is filtered thus separating the product from water or aqueous-alcohol soluble substances. The crude sapogenin may be purified by crystallization from hot heptane or by acetylation with acetic anhydride followed by crystallation from this solvent. A procedure useful with aqueous extracts is based on researches conducted by Krider and Wall (6) who found that whereas the steroid to sugar linkage is resistant to acid hydrolysis, the sugar to sugar bonds are easily cleaved with very dilute acids. The resultant steroid monoglycosides are water insoluble, may be collected by filtration or centrifugation, and then treated as described above. Alternatively the ground root may be treated directly with hot, aqueous acid, the insoluble solids collected, washed, and dried. Diosgenin is obtained by extraction of the solids with heptane, followed by crystallization.

Diosgenin is not an item of commerce in the sense that it may be readily purchased. Mexico places a prohibitive export tax on root, root extracts, diosgenin, and 16-dehydropregnenolone. There is no tax on pregnenolone, progesterone and other "finished" hormones. If it were available, diosgenin would probably sell in bulk from 5-10 dollars per pound. As will be shown in a later section diosgenin is a key source for progesterone and compound S which in turn are sources for corticosteroids via fermentation hydroxylation at C_{11}.

Hecogenin

Occurrence. Hecogenin, the structure of which is shown below is a 12-oxygenated sapogenin which



occurs in the leaves of many Yucca and Agave species (see reference 4 for detailed compilations). The only commercial source, however, is the leaf of *Agave sisalana* or the closely related *Agave foucroyedes*. Although the hecogenin content of the above species is low (0.1-0.2% dry basis), large quantities are available because *A. sisalana* and *A. foucroyedes* are grown in enormous tonnages as sources of sisal and hennequen fiber respectively. The Agave species are natives of the Western hemisphere. However they have long ago been exported to Java and East Africa. A typical hennequen area near Tampico, Mexico is shown in figure 2.

Preparation

Hecogenin is a by-product of sisal or hennequen production. Large tonnages of leaf are collected daily and brought from the fields to decorticators. The decorticators are large rotating drums with specially shaped surface areas which can macerate the leaf structure without breaking the fibers. The macerated, wet pulp and juices which ordinarily would be discarded are collected. The pulp is squeezed or drained and all the juices collected in large tanks. These juices contain the water soluble saponin, heconin. It

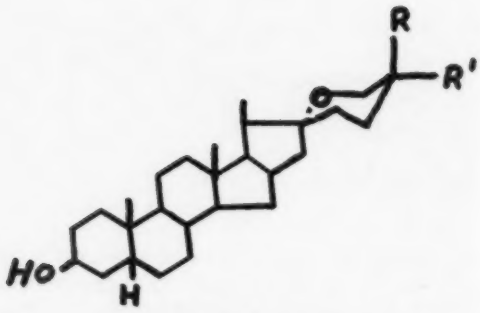
Figure 2. The writer standing in a group of mature Agave Foucroyedes plants.

is necessary that the sapogenin be recovered economically from very dilute solution. This can be done in several ways. In typical commercial processes developed at East African sisal plantations the dilute sisal liquors are allowed to stand in large vats for some time, often a week or more (7) (8). Often a vigorous fermentation ensues. The sludge which forms is a mixture of partially and completely hydrolyzed saponins. Studies at the Eastern Regional Research Laboratories have shown that native plant enzymes present in a variety of Agave species can hydrolyze saponins (9). Further researchers from this laboratory have demonstrated that a variety of microorganisms may develop adaptive enzyme systems which can produce hydrolysis of saponins (10). Undoubtedly the sisal sludge is formed by a combination of both plant and microbial enzymes. The sludge thus formed is collected by centrifugation after it has partially settled, and a large portion of the supernatant liquors is discarded. The centrifugation of the partially suspended sludge is most difficult. Filter cloths are rapidly clogged and a really adequate system still remains to be produced. The collected sludge is now further hydrolized with acid, taken up in hot heptane to which is added charcoal, filtered and allowed to crystallize. Often tigogenin is present as an impurity. Hecogenin is more insoluble in petroleum ether solvents than is tigogenin, so that a separation of the two is feasible by careful fractional crystallization. In the laboratory separation is also easily accomplished by chrometography (11).

Hecogenin is used almost exclusively for the synthesis of cortisone via an all chemical route. Glako Laboratories in England is the leading producer of cortisone from hecogenin, obtaining its raw material largely from East Africa and Mexico and at times from sisal grown in Jamaica and Haiti. Further details of the production of cortisone from hecogenin will be described in a subsequent section. Hecogenin is not a standard item of commerce. However companies like Syntex, S.A., Mexico City, and Root Chemicals, Roosevelt, Puerto Rico have supplied it in the past and raw material sources are unlimited.

Sarsasapogenin and Smilagenin

These two sapogenins are identical except for isomerism at C_{25} as shown below.



I R = CH_3 R' = H, Sarsasapogenin
II R = H, R' = CH_3 Smilagenin

Both steroids have the 5β-fusion at C_5 similar to bile acids. This type of fusion occurs much less frequently than C_5-α or C-Δ^5 fusions.

Occurrence

Sarsasapogenin occurs in the leaves of a number of Yucca species, many of them native to the Western United States. *Yucca bacotta* and *Yucca schidigera* are two particularly good species, containing up to 1.0% sarsasapogenin in the leaves, dry basis. A typical group of y. schidigera plants growing near Kingman, Arizona is shown in fig. 3. The lower leaves of

Figure 3. Yucca schidigera near Kingman, Arizona.

these plants may be harvested on a yearly basis so that repeated collections can be made from the same plant. Concentrated aqueous extracts of this species are being produced commercially in Kingman, Arizona and in Orange, California. The extracts are used in conjunction with water soluble fertilizers. Large but relatively sparse concentrations of *y. schidigera* are found in Northern Arizona and contiguous areas in California and Nevada.

Smilagenin is found in quantities of 1.0-1.5% dry basis in the leaf of *Agave lecheguilla*. This species occurs in enormous concentrations in southwestern Texas, centering in the Big Bend region. A typical group of plants is shown in fig. 4. In many areas it is considered a pest as it is poisonous to sheep due to a photosensitization process. The fiber in the leaf of *A. lecheguilla* has particularly desirable properties for use in brushes. Attempts at producing commercial fiber have failed to date because of the difficulties involved in the collection of wild material and the

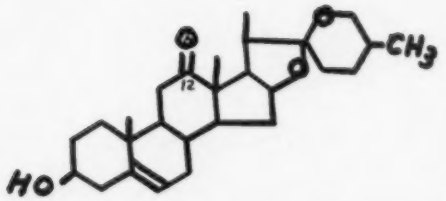
Figure 4. Agave lecheguilla, Big Bend National Park, Brewster County, Texas.

non-uniformity of such plants. Cultivation seems entirely feasible but to date has not received much study.

The processing of *Y. schidigera* and of *A. lecheguilla* for their respective sapogenins are identical. Extraction of the ground leaf or by-product pulps from fiber processing with hot water gives a saponin extract. Treatment with dilute sulfuric acid by the procedure of Kuder and Wall (6) yields quantitatively the insoluble partially hydrolyzed saponin. Purification via an alcoholic extraction of the crude saponin (11) followed by the stronger acid hydrolysis and heptane crystallization described under diosgenin, yields pure sarsasapogenin or smilagenin. Researches conducted at the Eastern Regional Research Laboratory have shown that these sapogenins can be converted to cortisone (12) via a common intermediate.

Gentrogenin (Botogenin)

This sapogenin has an unusual and most interesting background. The steroid has the structure shown below. It is thus 12-keto-diosgenin, combining in one



sapogenin the desirable chemical features of both hecogenin and diosgenin. In 1947, Marker and Lopez reported the discovery of a product with the 12-keto-diosgenin structure from *Dioscorea mexicana* (13) which they called "botogenin".

Upon the announcement of the anti-arthritic action of cortisone in 1949, an intensive hunt was instituted by a number of research groups for botogenin. Many samples of *D. mexicana* as well as other Dioscorea species were examined without success. Finally Dr. Howard S. Gentry of the Division of Plant Introduction and Exporation, USDA; who had been working in close cooperation with writer's group at the Eastern Regional Research Laboratory sent in a group of samples of *D. spiculiflora* for assay. D. spiculiflora is found chiefly in Chiapas province of Mexico near the Guatemalan border. The writer well remembers the excitement in his laboratory when infrared examination revealed the presence of the long sought 12-keto-diosgenin structure. Upon isolation of the sapogenin it was found that the physical properties differed enormously from those attributed to "botogenin" (14) (15). Hence we renamed the sapogenin, gentrogenin in honor of Dr. Gentry and another C_{25} isomer was called Correllogenin in honor of Dr. Donovan S. Correll another excellent botanist on the USDA team who also made several collection trips to Chiapas. The Eastern Regional Research Laboratory group has converted gentrogenin to cortisone by a variety of procedures (16) (17). The Syntex group has also

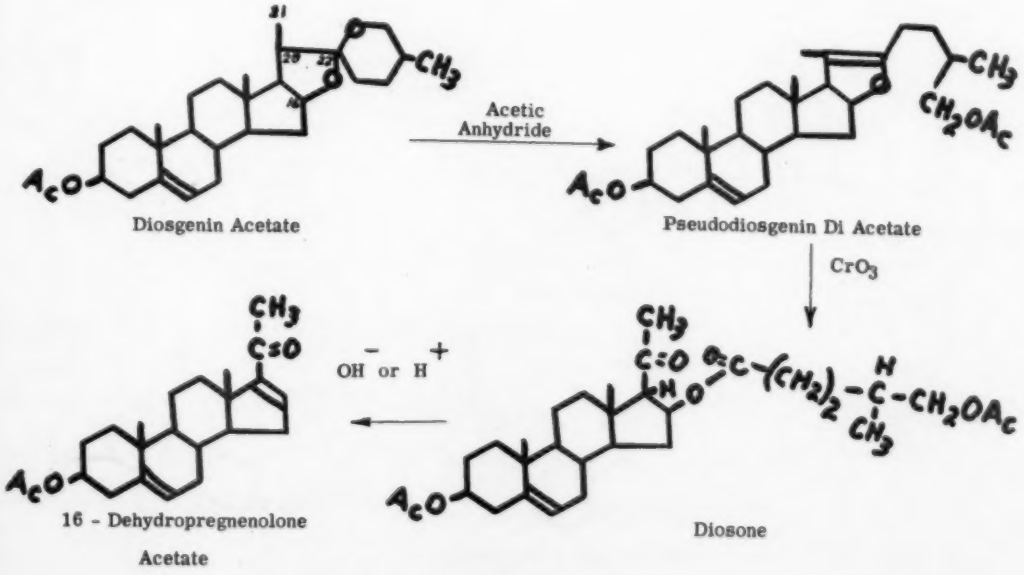


CHART IV

devised a cortisone synthesis from gentrogenin (18). The isolation and purification of gentrogenin has been described in detail (15).

Conversion of Sapogenins to Hormone Intermediates

The facile, three-step degradation of the Sapogenin sidechain to yield steroid hormone intermediates is largely responsible for the utility of steroidal sapogenins. The basic procedure is due to R. E. Marker and co-workers (2), (19) (20). The classical reaction sequence is shown in Chart IV illustrated by diosgenin. In Marker's original procedure, treatment of sapogenins at 200°C with acetic anhydride gave an unsaturated, delta$^{20(22)}$ steroid in which the spiroketal ring system was disrupted, ring F being opened. The resultant unsaturated compound called a pseudosapogenin on treatment with chromium trioxide underwent oxidation fission of the C_{20-22} double bond to give in the case of diosgenin, a compound called diosone. It will be noted that all of the carbon atoms except C_{20} and C_{21} which were once in Rings E and F are now part of a long chain ester at C_{16}. Alkaline or acidic treatment eliminates this ester forming a compound called 16-dehydropregnenolone. It will be noted that this compound has the C_{21} skeleton found in progestational or cortical hormones. A number of modifications and improvements have been made on the original Marker sequence (21) (22) (23) (24) (25). However no basic change has been made and this degradation sequence is undoubtedly one of the most important procedures used in steroid hormone technology.

16-Dehydropregnenolone is an extremely versatile steroid hormone intermediate. The versatility of this compound has become even more widespread with the advent of microbial hydroxylation at C_{11}, a technique pioneered by the Upjohn group (26). While it is beyond the scope of this article to review in detail the various syntheses of cortical, sex, and androgenic hormones from the basic 16-dehydro-intermediates, a few typical examples may be of interest.

Progesterone

Progesterone can be prepared from 16-dehydropregnenolone in two steps as shown below. Hydrogenation in the presence of a palladium catalyst saturates the delta^{16} without attaching the delta ^{5}-bond to give pregnenolone, another basic hormone intermediate which is commercially available. Oppenauer oxidation of pregnenolone gives progesterone. The procedure was used by Marker in the early 1940's. At this time progesterone was a rare and expensive chemical, worth about 100 dollars per gram. It is reported that Marker prepared about a kilogram fom diosgenin and thus immediately collapsed the entire price structure. At present progesterone in bulk quantities can be obtained for as little as 15 cents per gram. Progesterone is of course used as a progestational agent.

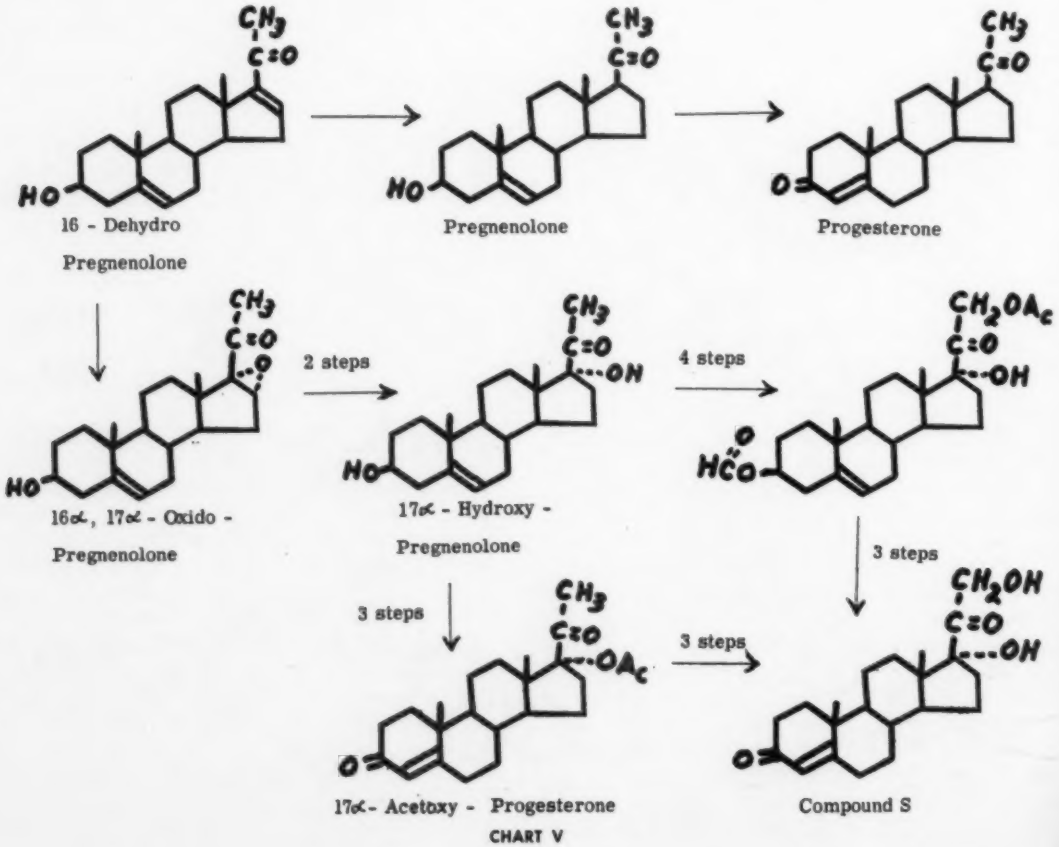


CHART V

Of greater industrial importance is the fact that progesterone is to date the best substrate for microbial hydroxylation at C_{11}. 11α-Hydroxy-progesterone which can be converted to cortisone or Hydrocortisone in a variety of ways (27) (28) (29) is another key bulk hormone intermediate. The introduction of microbial hydroxylation along with the availability of cheap progesterone was a large factor in reducing the cost of cortisone from $200 per gram to about $2.00. It should be mentioned, however, that improvements in the bile acid conversion to cortisone by the Merck group has allowed this process to remain competitive and thus also contributed to lowering the cost of cortisone.

Compound S.

Compound S while not useful for medicinal purposes is a key intermediate in the preparation of cortisone and hydrocortisone. The conversion of 16-dehydropregnenolone to Compound S can be carried out by a variety of interlocking procedures.[30] One of the best methods is shown in Chart V. Thus treatment of 16-dehydropregnenolone with alkaline hydrogen peroxide gives 16α, 17α-epoxy-pregnenolone which can be converted in two steps to 17α-hydroxy-pregnenolone. The above reaction sequence was developed by Julian and his associates.[31-32] Conversion of 17α-hydroxy-pregnenolone to the 3β-formate followed by the three-step introduction of the 21-acetate gives the 3-formate-21-acetate which is then converted to the 17-acyl-derivative. Oppenauer oxidation followed by saponification under mild conditions gives the desired Compound S.[33,34] Many of the compounds in this reaction sequence are themselves useful intermediates in particular 16α, 17α-oxido-pregnenolone and 17α-hydroxy-pregnenolone. As shown in Chart V, the latter can be converted via the 3-formate and 17 acetate followed by Oppenauer oxidation to 17α-acetory-progesterone, an oral progestogen.

Androgens and Estrogens

Steroidal Sapogenins via their 16-dehydro-pregnenes can be converted into a variety of androgenic and estrogenic steroids with the basic 19-carbon structure. While it is beyond the scope of this article to describe the chemical preparations of the many steroids in this classification, the basic procedure is shown below in Chart VI. Thus in a general procedure applicable to all 16-dehydropregnenolones,[35] the latter as converted to the corresponding oximes which undergo Beckmann rearrangement on treatment with p-acetamino-benzenesulfonyl chloride yielding an amide. The latter on acid hydrolysis gives the 17-betone. In this manner on a very large scale 16-dehydro-pregnelone acetate can be converted to androstenolone, see chart VI.

The basic estrogen, estrone can be prepared from diosgenin using the Beckmann rearrangement[35,36] as shown in Chart VI.

Anti-Inflammatory Drugs

Corticosteroids with anti-inflammatory action constitute the most important group of steroid hormones,

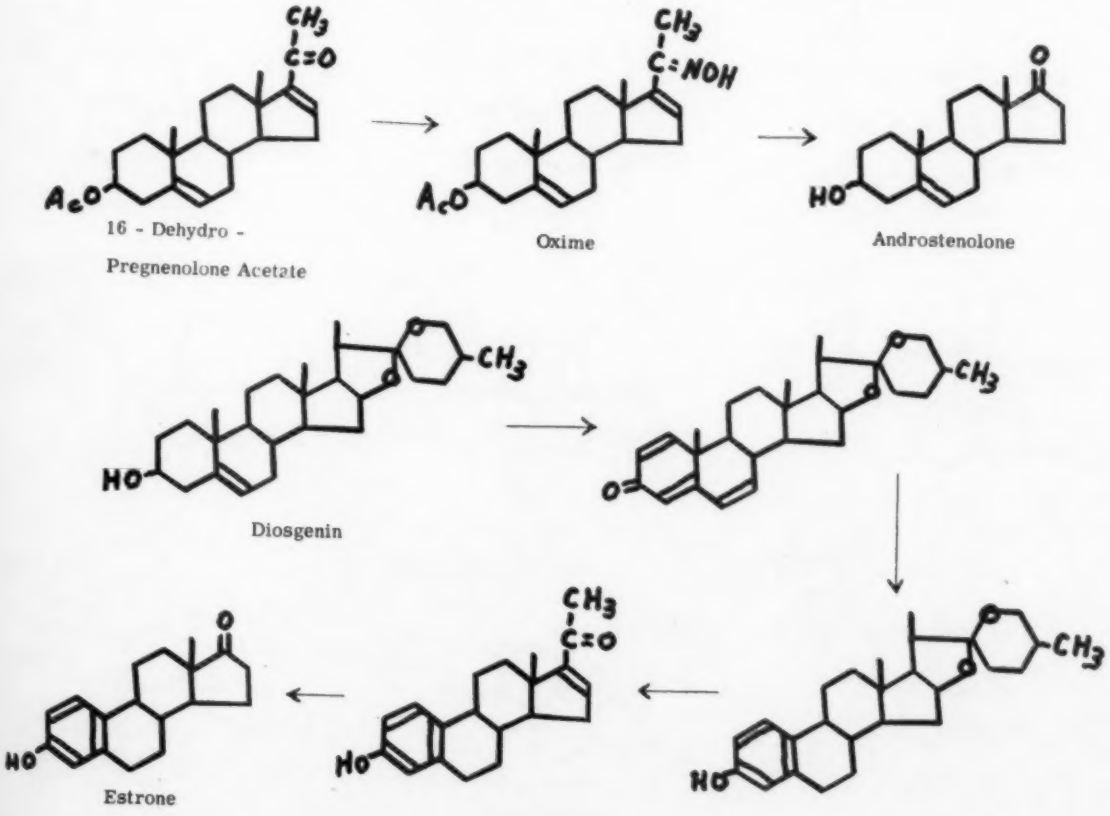


CHART VI

their market being in the neighborhood of 100 million dollars. Chart VII shows some of the more important anti-arthritic steroid hormones. After cortisone and hydrocortisone had been in use for some time subsequent to their introduction in 1949, undesirable side-effects became increasingly apparent, especially after long-term administration. In particular such effects as excessive sodium retention, edema, increased gastric acidity and psychosis became evident. Much research, most of it unsuccessful, was conducted with the hope of achieving a modification in structure which might result in eliminating the undesirable side effects without affecting the anti-arthritic action. The first breakthrough came as a result of the researches of the Squibb group, in particular J. Fried and associates.[37-38-39] These workers prepared the 9α-fluoro-derivatives of cortisone and hydrocortisone and found that they were about ten times more active than the corresponding non-fluorinated compounds. Unfortunately the new fluoro-analogues on administration caused a marked increase in sodium retention leading to edema. As a result 9-fluorohydrocortisone is at present used chiefly for the maintenance of patients with Addisons disease.

In 1955, the Schering group[40,41] made an important advance in anti-arthritic therapy. These workers prepared the 1-dehydro anologues of cortisone and hydrocortisone, calling them prednisone and prednisolone. They can be prepared by the action of Corynebacterium simplex[41] on cortisone or hydrocortisone. Dehydrogenation of the latter steroids with selenium dioxide also leads to the desired 1-dehydro-analogues via a chemical procedure.[42,43] Prednisone and prednisolone are much more active in their anti-arthritic activity than the parent compounds. As a result lower dosages are required and in general the deleterious side effects were materially reduced. The 9α-fluoro-analogue of prednisone or prednisolone as might be expected are considerably more potent than the parent substances. However the former are too potent in mineralcorticoid activity to permit their use in disease except by topical application.

In a series of investigations first reported in 1955, Bernstein and co-workers at Lederle (American Cyanamid) discovered that 16α-hydroxy-cortisone had considerable glucocorticoid (correllated with anti-arthritic action) and little sodium retention.[44,45] As was now becoming standard practice, the Lederle group then synthesized a compound containing the 9α-fluoro and 1-dehydro-moieties combined with the 16α-hydroxy group.[46] The 9α-fluoro-16α-hydroxy-1-dehydro-hydrocortisone product is marketed under the trade name Triamcinolone. It is a potent anti-arthritic agent and moreover, due to the presence of the 16α-hydroxy group is almost entirely devoid of salt-retaining properties.

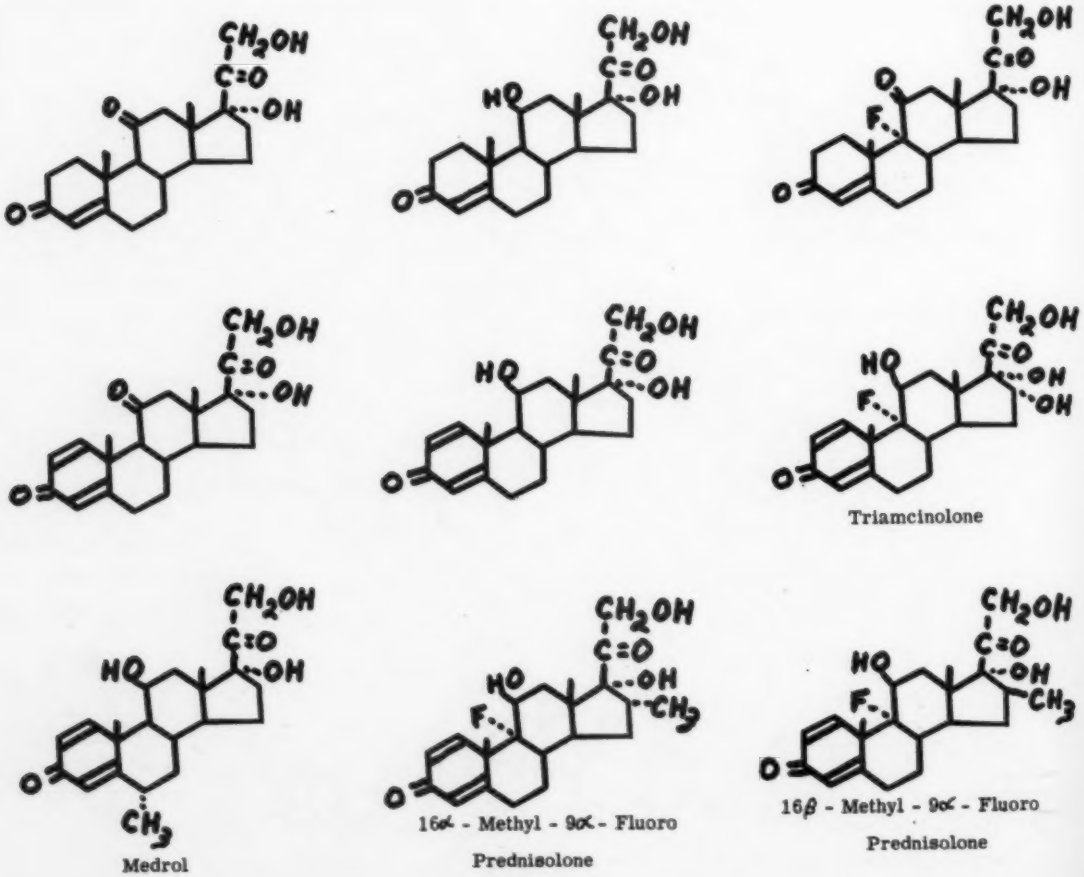


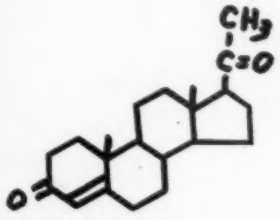
CHART VII

Another modification in cortical hormone structure was found by the Upjohn group.[47,48,49] Working first with the 2α-methyl cortical analogues and then with the more favorable 6α-methyl derivatives, the Upjohn group prepared 6α-methyl-9α-fluoroprednisolone and 6α-methylprednisolone. Both are active anti-arthritic agents with little salt retention or edematic activity. The latter is somewhat more satisfactory and is marketed by Upjohn under the name, Medrol.

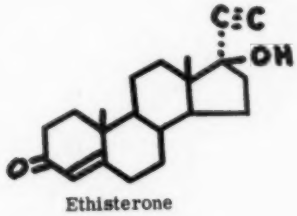
Another potent methylated anti-arthritic steroid was prepared almost simultaneously by the Merck and Schering groups. These were the 16α-methyl and 16β-methyl-9α-fluoro-prednisolone.[50,51,52,53] The former is the most potent non-salt retaining anti-arthritic steroid reported to date. It would seem certain, however, that many more analogues combining every possible feature are being actively studied. A full description of the various cortical analogs is given by Fieser and Fieser (reference 3, pp. 682-708) and also in a recent review by Fried and Borman.[54]

Progestational Agents

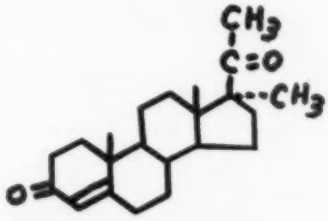
The earliest steroid with progestational activity was isolated from the corpus luteum[55] and characterized[56] between 1930-1934. This hormone was called progesterone. As we have noted in an earlier section, progesterone is made on a large scale from diosgenin. Progesterone can also be produced efficiently from stigmasterol.[57,58] Stigmasterol occurs in soy bean oil and is isolated in a "foots" fraction during purification of the oil. It is always found in conjunction with sitosterol. The separation of the two sterols, which was formerly difficult and inefficient, has been effectively solved by the Upjohn group.[59] Although there was an active search for other progestational agents only one other really active product, ethisterone (17α-ethynyl-testo-sterone)[60] was discovered in the period 1937-1950. In 1950, the highly active 17α-methylprogesterone was found by a Swiss group.[61,62] Several years later 17α-acetoxyprogesterone became available[63] and was found to possess high activity on oral administration. More recently the techniques applied to the formation of cortisone analogues, described in a previous section, have been applied with marked success to the progesterone field. Thus 6α-methyl-17α-acetoxyprogesterone[64] and 6α-fluoro-17α-acetoxyprogesterone[65] have enormously high potencies. Introduction of a double unsaturation in ring A as in prednisolone gives even greater activity. Progestational activity may also be increased by removal of the angular methyl group at C19 to give 19-nor-steroids. Thus 19-norprogesterone[66] and 19-norethisterone[67] (Norlutin) are potent progestens as is also the delta$^{5(10)}$ isomer of the latter (Enovid).[68] Both norethisterone and its delta$^{5(10)}$ analogue prevent ovulation and show promise as oral contraceptives. Chart VIII gives the structure of the various progestational hormones described above.



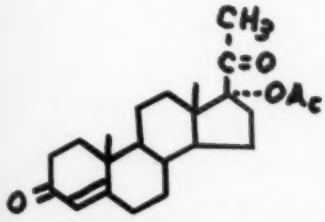
Progesterone



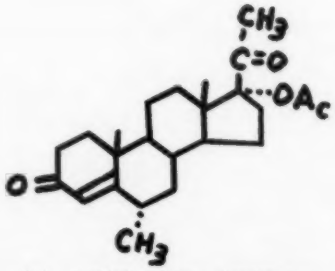
Ethisterone



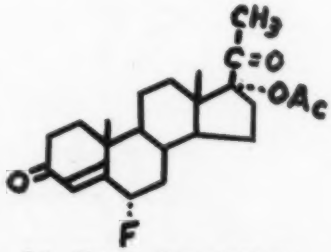
17α - Methyl Progesterone



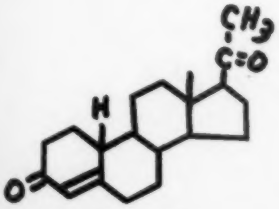
17α - Acetoxy Progesterone



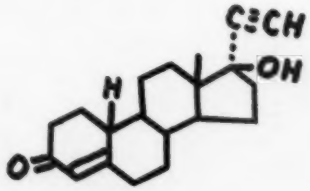
6α - Methyl - 17α - Acetoxy-Progesterone



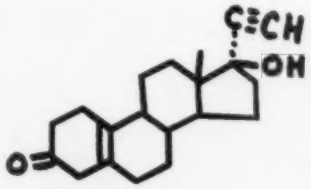
6α - Fluoro - 17α - Acetoxy Progesterone



Nor Progesterone



Norethisterone



Enovid

CHART VIII

Estrogens

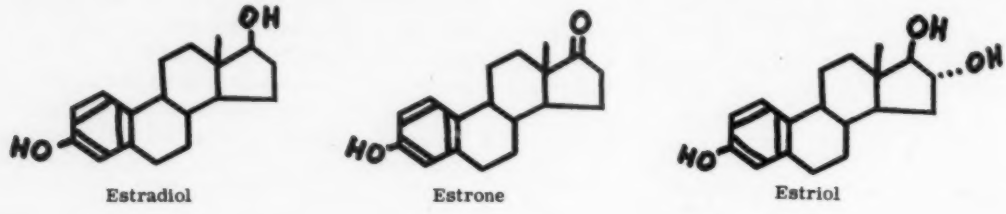
An important group of female sex hormones, responsible for estrus, is called the estrogenic hormones or estrogens. Chart IX gives the structure of a number of the more important estrogens. Estradiol is produced in the ovary and is excreted in the urine along with its metabolites, estrone and estriol which are much less active subcutaneously. However, estriol is more active orally than estradiol. Equilenin and equilin are other members of this class of naturally occurring hormones. Estrone can be converted to 17α-ethynylestradiol[69] which is a potent estrogen both by injection or orally.

The entire group of estrogenic substances is readily available from sapogenins, particularly diosgenin. The process for this conversion has been given in an earlier section, see chart

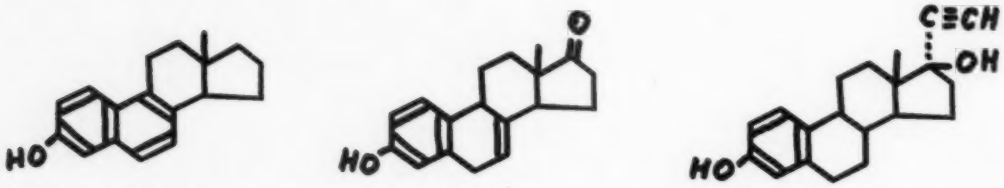
Estrogens are chiefly used in cases of estrogenic deficiency. Minor uses are for carcinonea of breast and prostate, in veterinary medicine, and in cosmetic formulations. Estrogens are absorbed through the skin. However, there is some question as to the significance of this absorption.

Androgens and Anabolic Agents

The group of steroid hormones found in the testes are called androgens and are the male sex hormones. Some of the more important representatives of this class are shown in chart X. The earliest discovered

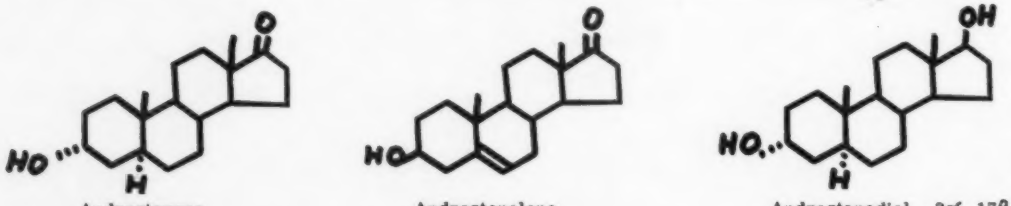


Estradiol Estrone Estriol

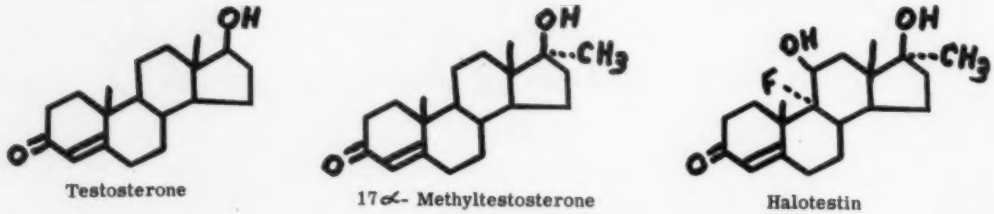


Equilenin Equilin 17 - Ethynylestradiol

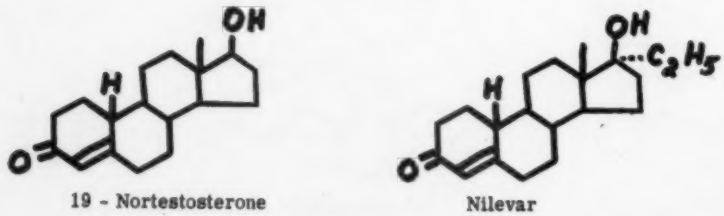
CHART IX



Androsterone Androstenolone Androstanediol - 3α, 17β



Testosterone 17α- Methyltestosterone Halotestin



19 - Nortestosterone Nilevar

CHART X

potent androgen was androsterone.[70] Androsterone is now readily available from androstenolone, a compound easily prepared by degradation of cholesterol or diosgenin (see chart VI). Androstanediol-3α, 17β[71] prepared by reduction of androsterone is three times more potent than the latter. An even more active androgen, testoslerone was discovered in 1935.[72] Testosterone can be prepared from androstenolone by a variety of methods.[73] 17α-methyltestosterone[74] is another active substance and is particularly useful in oral therapy.

In addition to their androgenic properties many of the male sex hormones exhibit another physiological effect, classified as protein anabolic activity. An anabolic agent promotes protein storage and is a general tissue stimulant. Hence it soon became apparent that if the androgenic properties could be minimized by structural variation without affecting anabolic activity, useful drugs for treatment of debilitated or aged persons would be obtained. 19-Nortestosterone[75] represented a first advance. However, 17α-ethyl-19-nortestosterone (Nilevar)[76] has high anabolic activity and only slight androgenicity. Another active anabolic agent with low androgenic properties is 9α-fluoro-17α-methyl-delta^4-androstene-11β, 17β-drol-3-one (Halotestin).[77]

Miscellaneous Uses

It is beyond the scope of this article to go into the many uses, actual and potential for steroids which have not been covered in the previous discussion. A few uses which may be briefly mentioned are:

(1) *Anticancer drugs.* Testosterone and estradiol were used as palliatives in cases of carcinoma of the prostate and mammary glands. A host of newer nonfeminizing estrogens and nonmasculinizing androgens are under test.[78]

(2) *Cardiovascular agents.* This group is one of the largest potential markets for steroid drugs. Several representative steroids are 16-chloroestrone methyl ether (antiatherosderotic), 16-cyclohexylamino-allopregnandiol (antiarythmic), 17α-propyl-4, 5-dihydro-19-nortestosterone (hypotensive).

(3) *Agents acting on central nervous system.* Examples of steroids with this type of activity are dihydrodesoxycorticosterone sodium hemisuccinate (Viadril), pregnenolone, and 6β-hydroxy-3, 5-cyclopregnan-20-one (Neurosterone).

(4) *Veterinary Products.* Use of steroids to improve feed utilization, regulate breeding cycles or to control milk production has enormous potential. In present use are progesterone or testosterone combinations with estradiol, 17α-hydroxyprogesterone, and many of the newer non-androgenic anabolic steroids are being tested.

(5) *Cosmetics.* Steroids are being tested for prevention of baldness in men and hirsutism in women. Creams containing pregnenalone and progesterone are being marketed as an aid to proliferation of skin layers and for "plumping" of skin tissue. Preparations containing estrone and estradiol have been on the market for some time in skin creams.

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Solubilizing perfume oils in aerosols

A process has been developed by Salvatore Noto in the laboratories of Sun-Lac, Inc., that is claimed to solubilize any and all perfume oils in fluorocarbon propellants. The process does not add or subtract any material. Instead, it consists of a technique of mixing the oil, alcohol and propellant, under the right conditions of temperature and pressure.

This development is very significant in view of the formula variations to which many perfume houses have had to resort, in adapting their well-known perfumes to aerosols. This has been due to the incompatability of many essential oils and aromatic chemicals when mixed in the normal manner with the fluorocarbons. Many references in the literature have noted these incompatabilities, resulting in both precipitates and loss of certain fractions of the perfume, particularly the top notes and the fixative.

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The precipitates formed in the loading of conventional perfume oils in aerosols is exceptionally fine, often taking several days to several weeks to appear after the loading takes place. In fact, at least a portion usually does not settle, but remains as a cloud or layer. However, the most important difference between this type of precipitate and that normally encountered, and filtered out, in the compounding of perfume oils, concerns the perfume itself. Whereas the compounding precipitate is composed of non-aromatic fractions, compounds that formed due to the presence of the fluorocarbon propellants contains some of the vital aromatic substances and the resulting perfume lacks a significant part of its total effect.

As an indication of the effectiveness of this new process, the following perfume formula, containing many materials reported incompatible with fluorocarbon propellants, was very successfully solubilized.

BOUQUET S-119

21.25	OIL BERGAMOT NATURAL
7.50	OIL PETITGRAIN S.A.
7.50	VETIVERT ACETATE
6.25	• MUSK AMBRETTE
6.25	ISO EUGENOL
5.00	AMLY CINNAMIC ALDEHYDE
5.00	• CUMARIN
5.00	BENZYL ACETATE
5.00	NEROL
5.00	ALDEHYDE C-12 MNA-10% IN DI ETHYL PHTHALATE
3.75	OIL YLANG BOURBON
3.75	•• RESINODOR OPOPONAX
5.00	•• JASMIN ABSOLUTE
3.00	OIL PATCHOULI
2.50	•• OAKMOSS RESINEX
2.50	•• RESINODOR BALSAM PERU
1.25	OIL LAVENDER 40-42%
1.25	• VANILLIN C.P. USP
1.25	•• RESINODOR STYRAX
1.25	STYRALLYL ACETATE
0.75	OIL TARRAGON

• Dissolved solids—high % will precipitate

•• Materials have been mentioned in articles as incompatible

Conventional formula— 1.5 above perfume
13.5 alcohol 39c
15.0 propellant 114

This oil was compounded into an aerosol by the following formula: 1.5 parts S-119 Bouquet, 13.5 parts Alcohol 39c, and 15.0 parts Propellant 114. This formula formed a noticeable precipitate when loaded in the conventional manner, after about 48 hours. When solubilized with the new process it remained clear and stable after six months.

Salvatore J. Noto, chief chemist at Sun-Lac, Inc., is one of those rare technical men who combines substantial experience in more than one field. He is a former perfumer with the Aromatics Division of Verona Chemical Co. He got his start in the aerosol field as a consultant with Fluid Chemical Co., and later helped Fluid set up its first perfume aerosol line. Sal has been with Sun-Lac for four years where he is responsible for new product development and quality control.

Bulgarian essential oil

From Chamber of Commerce
Sofia—Bulgaria

Bulgaria holds a leading place among the countries producing essential oils, as a natural consequence of the qualities of her essential oils. It can safely be asserted that many foreigners think about Bulgaria in terms of the country's attar of rose, and this again has a bearing on its unique qualities.

One of the principal factors which account for the high quality of Bulgarian essential oils are the country's favourable conditions of soil and climate, particularly in the regions where the essential-oil crops are raised. For instance, the attar-yielding rose (Rosa damascena Mill.) thrives best in the Bulgarian Rose Valley, irrespective of its origin. This is a strip of land stretching between the Balkan Range and the Sredna Gora Mountain, embracing the Levskigrad and Kazanlik Plains and the southern slopes of the Sredna Gora. The Levskigrad Plain, the valley of the Strema River in particular, proved a most favourable place for the cultivation of Mentha piperita which yields the famous peppermint oil of Bulgaria.

There is yet another factor determining the high qualities of the Bulgarian essential oils, in addition to the favourable climatic and soil conditions of the country. This is the manner in which they are obtained. In recent years these oils have been obtained in modern industrial installations supplied with up-to-date appliances and instruments. The technological processes applied are based on the results of studies made on the elements determining the yield and qualities of Bulgarian essential oils.

The Bulgarian attar of rose has long enjoyed a high reputation among specialists in the perfumery industry. It is an acknowledged fact that, due to the fundamental properties imparted to perfume compositions by the Bulgarian attar of rose, it has become an indispensable element of many high-grade perfumes. By its properties, well known both in the past and now, the Bulgarian attar of rose holds first place in the world.

Bulgarian peppermint oil, known under the name of "Bulgaro micham" is obtained from Mentha piperita. It has a typically fine aroma and a pleasant cooling taste. These qualities assign it a place of honour among the best peppermint oils in the world. Bulgaria produces two types of peppermint oil—raw and rectified.

The Bulgarian oil of lavender is obtained from the blossoms of Lavandula vera and possesses properties which make it a most original material for the perfumery industry.

The coriander oil of Bulgaria is rich in linalol whose content reaches 58.3 per cent. This is much higher than the linalol content of coriander oils obtained in other countries.

The Bulgarian sweet-basil oil possesses a strong and original aroma. Bulgarian anise-oil contains a high percentage of anethol. It has a pleasant taste and fine aroma.

Wild-geranium oil is a valuable raw material for the perfumery industry. It adds stability and freshness to the perfume compositions accompanied by the tone of the musk.

The oil of Salvia sclarea obtained in Bulgaria has a high content of linalilacetate which may reach up to 78 per cent.

The Bulgarian oil of camomile has most valuable properties—a typically fine and full aroma and a dark-blue colour. Its azulene content reaches 6 per cent.

The manufacture and sale of essential oils is under the control of the state. Most of the essential oils produced in the country are exported abroad where they find a favourable market.

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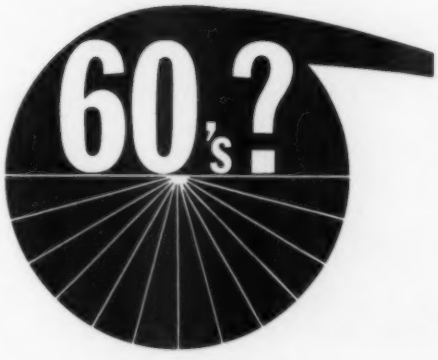

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BRANCHES THROUGHOUT THE WORLD

PACKAGING & PROMOTION

1

1—Bonne Bell

Each package of Bonne Bell's "treatment Trio" contains a recording by Bonne of a personal message of beauty advice, outlining the importance of each product in the package, and how to use it. The new package combines three of her most popular beauty preparations, "1006" "Medi-Masque," and "Medicated Make-Up". The novel feature of the recording promises to enhance its sales appeal, especially with the teen-age market.

2

2—Positan

The latest entry in the sun-tan field is a dual-action lotion which prevents burning in sunlight and also produces a tan without the sun. A companion product to Man-Tan After Shave Lotion, Positan was brought out when it was discovered that the shave lotion was being used by both men and women on arms and legs as well as on their faces to produce a sun-tan appearance.

Packaged in four ounce and eight ounce containers, Positan, is available in clear or creamy lotion. The clear lotion is marketed in plastic squeeze bottles and the cream lotion in glass bottles.

3

3—Tangee

For the first time in its history the House of Tangee is producing a full line of nail polishes. Eight regular shades and five iridescent shades are being introduced this month in retail outlets. The polish features Protogel an ingredient promoted to help nail growth and resist chipping and splitting.

An advertising program featuring full-page color ads in national magazines will introduce the new line, starting in May.

4—Coty

Following research which showed that women prefer to carry two lipsticks, Coty has come out with a tortoise shell case with a lip size mirror on top and containing two Coty "24" lipsticks. Called the Riviera Twin Lipstick Case, the item will retail at $2.25.

Several coordinated color combinations will be available but the consumer will be able to select her own combination of colors since any of the full range of Coty "24" lipsticks will fit into the case.

4

5

6

5—Old Holland

A combination of beer, coconut oil and lanolin based on a formula developed by Harry Kay has produced Old Holland Beer Shampoo, a product of Silver-K Corporation, Manchester, New Hampshire. The shampoo is being introduced nationally in drug and chain stores in a four-ounce and a seven-ounce size. Recommended for the whole family, the shampoo is being promoted as giving body and sheen to the hair while making it easier to manage.

6—Mennen

Mennen's Electric Pre-Shave, Prop, is being marketed in a clear glass bottle molded in a shape suggestive of an electric shaver and capped with a specially designed white twist type cap which suggests an electric shaver guard. The unique cap is the only one of its kind in the country and was designed to Mennen specifications. It fits over a snap plug which makes the bottle an applicator container.

7

7—Helene Pessel

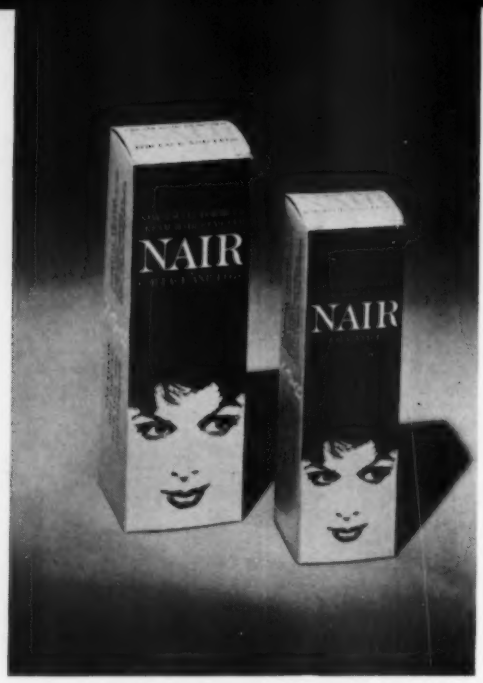
Helene Pessel, Inc. has just announced the introduction of a "Little Lady" Counter Display unit, prepacked with an assortment of nine of the fastest selling items in the line. It provides a *complete* gift department for little girls, featuring a wide variety of inexpensive scented accessories. Each item is on a plastic platform and the whole is enclosed underneath a clear, heavy-duty acetate cover. The display can be kept clean and is proof against pilferage.

8

8—Max Factor

A new Hand and Body Smoothing Lotion has been introduced by Max Factor which is claimed to be different from anything in the market, and is scented with Max Factor's two topselling fragrances—Hypnotique and Primitif. This new lotion contains special healing and soothing ingredients, plus newly perfected fast-acting emollients. It is claimed beneficial even to the most sensitive skins, and not only relieves chapping and roughness, but continued use prevents recurrence of these conditions when skin is subjected to the climatic extremes of cold, dampness, drying wind, and heat. Each version is packaged in the same distinctive design motifs of the fragrance lines, with black capped gold labelled bottles for the Primitif scented Hand and Body Lotion and glittering textured gold label packaging for the Hypnotique scented Lotion.

The judges thought it would give the housewife a lift to pick one of these Drene packages off a shelf. Designed in various shades of pink, the cartons are printed in five colors and lacquer.



An unusual face bearing a provocative look gives these new cartons striking shelf appeal. This design replaces a black and white photograph that formerly appeared on these packages.

This Revlon carton won its award by its very bright color and the picture of the dancing girl. The judges declared it the type of package any woman would be inclined to buy.

Merit award winners—in the folding paper box association annual competition

When we found that no first prize was given in the cosmetic category at this competition, we asked Mrs. May Bender, one of several judges in this category, and a package designer, to give us some of the reasons why the judges withheld a first award. The following are her notes:

"When one thinks of cosmetics, one thinks of glamour, color, excitement. And the cosmetic package should be synonymous with the product. After all when a woman buys beauty in a box, the box must be beautiful too. Obviously, the judges felt that merchandise-wise, the best was nowhere in sight.

"It is well-known that a great deal of money is spent on cosmetic packaging, however the majority are set-up packages. Perhaps that is why there were no first prize awards. Even the merit awards were hard come by.

"Maybe this situation occurred because most of the entries were geared for supermarket and variety store consumption and few were directed at more expensive purchases. Maybe the manufacturer still believes that a folding box does not connote quality and therefore steers away from them, and maybe it was simply that there just was a dearth of entries in cosmetics.

"I do not mean to diminish the entries and certainly not those which rated merit awards, which were fine examples of the printed art and paper-makers and embossers art. It's just that there is room for more scope than was evident among the entries.

"I do believe that manufacturers and perhaps designers are missing the boat when they do not use imagination in folding box design. Certainly many elaborate and interesting and unusual cartons can be devised."

The well-established word "Ivory" is prominently displayed. The red, white and blue colors, with a touch of gold, connote family use of the product instead of being strictly a feminine item. An interesting additional feature is an outlin eof the shampoo bottle embossed on the front.

The daisy design and the reverse type on bright blue foil were particularly appealing to the judges. They found this whole family of closely related Pharma-Craft cosmetic items unusually attractive.

TECHNICAL ABSTRACTS

An analysis of the Volatile Oil of Lomatium Suksdorfii Frank A. Pettinato, Louis Fischer, and Nathan A. Hall

The volatile oil of the fruits of Lomatium Suksdorfii (Wats.) Coulter and Rose, obtained by steam distillation, was found to consist of over 90 per cent terpene hydrocarbons among which α-and β-pinene, *d*-limonene, and *d*-β-phellandrene were identified. Isovaleric acid was identified as a component of the oil, and both acetic acid and isovaleric acid were found in the aqueous portion of the steam distillate. *Thru J. Am. Pharm. Assoc. Sci. Ed., 49, 1, 45(1960).*

The Preparation and the Properties of an Aspirin Homolog β (o-Acetoxyphenyl) Propionic Acid—Charles W. Bauer and Edward Francis LASALA

A homolog of acetylsalicylic acid, β-(*o*-acetoxyphenyl) propionic acid, has been made successfully. Its toxicity and, analgesic properties have been determined on rats. Its oral LD_{50} on rats is over 1.5 mg/Kg. It has been found to have analgesic properties in rheumatic patients. No free salicylic acid is eliminated in the urine. It appears that this compound does not exert its analgesic properties as a salicylate. *Thru J. Am. Pharm. Assoc. Sci. Ed., 49, 1, 48(1960).*

Over the Autoxidation of Emulsified Fats, Prof. Dr.-Ing. M. Loncin and Dr.-Ing. D. Jacqmain

The author reports that in case of emulsified fats the speed of autoxidation is dependent upon the heavy metal, especially copper content in the oil phase. The transference of copper from the oil to the water-phase leads to the stabilization of the fat and is brought about by the complex formation with the help of alanin. The process can be exploited practically. The experiments show that the storage life of dry milk can be enhanced 3 to 4 times in this way. The stability of butter is however not affected to the same extent. *Thru Fette. Seifen Anstrichmittel, 61, 10, 1055 (1959).*

Determination of Adhesive Power of Cosmetic Powder Constituents, Dr. H. Neugebauer and Dr. S. Gruner, Baden-Baden

For the characterization of adhesive properties of powder or its constituents a new method has been developed by modifying the Jacobi Method. It is simple and the margin of error is also relatively small. The surface for carrying out the experiments is provided by a wild leather type plastic—YAK, which can be compared to the human skin so far as the adhesive characteristics are concerned and gives reproducible results. The determination of remission is carried out by photometry and the formula through which the adhesive power is derived is given. *Thru Fette. Seifen Anstrichmittel, 61, 1, 994,(1959).*

Physical Properties of Lipids Used in Pharmacy I—Screening Raw Materials Via Photomicrography, Davis R. Reese, Clifford W. Chong, and Joseph V. Swintosky

A simple photomicrographic technique has been devised to screen lipids for integrity of their crystal structure. In this technique, thin films of the materials are prepared on microscope slides and photomicrographs are taken of their surface and internal structures. The samples are then stored at normal or exaggerated temperatures for any desired period of time. At various intervals, the exact areas appearing in the original photomicrographs are rephotographed and compared with the originals. These permanent records permit detection of subtle changes which occur during aging. The technique has been used with a number of individual materials and simple mixtures. Representative photomicrographs are presented showing the structural integrity of carnauba wax, cetyl alcohol, hydrogenated soya oil, lauryl monoethanolamide, stearyl alcohol, and white wax. *Thru J. Am. Pharm. Assoc. Sci. Ed., 49, 2, 85(1960).*

Dermatherapeutic Aspect of the External Application of Cortisone, Dr. R. Haensch

In clinical experimental investigations the Cantharidine Test has been found to be more suitable than the Ultraviolet-Rays-Erythem and Histamine-Quaddel-Test for the detection of Prednisolon-effect. The author is of the view that the use of an ointmentbase, which should be suitable for the nature and constitution of the skin, would result in increasing the therapeutic effect. In case of treatment of contagious exzemas, the endogen exzema, the so-called seborrhoic exzema and various dermatitis forms two emulsions (o/w and w/o with the addition of Neomycin) containing Prednisolon are used. *Thru Fette. Seifen Anstrichmittel, 61, 10, 989(1959).*

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Technical digests

The Functions of Humectant in Vanishing Creams.
By D. M. Bryce, F.P.S., and J. K. Sugden, M.P.S.

(digested from PHARMACEUTICAL JOURNAL, p 311, Nov. (1959).)

Polyhydric alcohols have been used in pharmaceuticals mainly for their solvent abilities and their osmotic and humectant effect in high concentrations. Their effect in low concentrations in vanishing creams has been studied by Bryce and Sugden in an effort to evaluate the resulting product as a foundation cream. There have been many investigators examining this problem. Harry, in 1944, used humectants to maintain the water content at a normal level when the atmospheric conditions varied. Griffin, Behrens, and Cross used glycerin, propylene glycol, and sorbitol in their work and found glycerin to be the most hygroscopic under equilibrium conditions. De Navarre showed that the polyols played a direct part in the emulsifying system. Evanson, Henney, and Sperandio concluded from their experiments on relative humidities that although sorbitol appeared to be the best all-round humectant for cosmetic use, the best selection of a humectant was based on an accurate knowledge of the humidity conditions that would be encountered. The influence of a humectant on the various properties of a simple vanishing cream was recorded and discussed. In assessing the degree of drying out, the work was done at the ambient humidity and temperature of a large factory. The cream was made to the following formula:

Stearic acid B.P.C.	22%	w/w
Triethanolamine B.P.C.	1%	
Methyl p-hydroxybenzoate	0.2%	
Distilled Water	up to 100%	

The humectants, glycerin, propylene glycol, 70% sorbitol syrup, and polyethylene glycol 400 were incorporated into the cream. A series of creams containing 1, 2, 4, 8, 16, and 32 per cent w/w of humectant was produced, and the creams were packed into weighed polystyrene jars. Their weights were observed over a thirteen day period. Throughout the series studied, one per cent concentration of humectant exhibited remarkable properties of water retention which could be the result of the crust formation at this concentration. It was noted that with the exception of sorbiol creams all the water losses from the 2, 4, and 8% creams were similar. The turning point was at about the 2% level. Consequently, this phenomenon brought about the hypothesis that there was a modification of the properties of the emulsifier system. There was an observation of the cooling curves of the components of the emulsion system severally and in combination. The effect on these curves of the relevant concentrations of humectants was observed. It was concluded a change in the system had occurred.

Thus, these conclusions followed:

1. A polyol added to an emulsion may affect its properties.
2. There is a critical concentration of humectant above which further additions of polyol leads to a decrease in the water-retention capacity, but a massive addition reverses this trend. The critical concentration has to do with the part the polyol plays in the emulsion.
3. Sorbitol in a humectant soap-complex did not improve the water-retention properties of the cream.
4. Crust formation can be reduced in all cases by the use of a humectant.

A Note on Gravimetric Estimation of Vanillin Via Its Semicarbazone.
By K. K. Kaistha.

(digested from DRUG STANDARDS, volume 27, No. 5 Sept.-Oct. 1959.)

This method for the estimation of vanillin comes as a result of the successful determination of camphor via its semicarbazone by the same worker in 1958.* Vanillin has been estimated by titrating an alcoholic solution of the sample with alcoholic potassium hydroxide. Other methods include using dinitrophenylhydrazine gravimetrically. The method described here uses semicarbazide hydrochloride. The vanillin was dissolved in 80% aldehyde-free alcohol and refluxed after the addition of semicarbazide hydrochloride and potassium acetate. The precipitate, collected and dried in a tared Gooch crucible, is equal to 0.7271 Gm. of vanillin. The estimation of vanillin was done by the three different methods suggested by Sharpe and the British Pharmocopoeia, 1948 and 1953. The table representing the results of all four methods showed that Method I (semicarbazone) produced results that were not high and had good precision as long as the exact procedure was followed. The other gravimetric determinations produced inconsistent and big results. The volumetric method is not satisfactory because of the end point.

**Kaistha, K. K., DRUG STANDARDS, 26, 83 (1958).*

The Water Absorbability of Petrolatum Ointment Bases.
By Gordon A. Groves, and D. J. G. De Boucier.

It has been shown that there is a maximum concentration of Span which may be used in order to form an absorption ointment base with petrolatum. Petrolatum has its disadvantages that include a greasy character and poor hydrophilic properties. Newman and Miller used non-ionic surfactants in concentrations ranging from five to ten percent in order to increase the washability of some absorption bases. The study by Groves and De Boucier, as reported in the *CANADIAN PHARMACEUTICAL JOURNAL*, Vol. 93. No. 1.,* is an attempt to develop a system of formulation with which an ointment could be expected to absorb a pre-determined amount of water.

The method consisted of triturating a 20 gram portion of the petrolatum ointment base, including the addition of either the selected Spans, Tweens, or Carbowax 1540, with water in 0.5 cc. portions. When no more water was taken up, the mixture was chilled and levigated on a slab. If the mixture exuded water, the process was repeated using 0.1 cc. less water. The amount of water absorbed was recorded in both the number of ccs. and by the "water number". Petrolatum alone had a water number of 6.0 (20 gms. of petroleum absorbed 1.2 ml. of water). The Spans were more effective than the Tweens in absorbing water. In particular, Span 80 had a water number of over 150, and it was shown that this value could be obtained with a surfactant concentration of 1.5%. It was also noted that a concentration exceeding 1.5% displayed a curiously decreased ability to absorb water. Carbowax 1540 showed relatively little potentiation on the water number of the petrolatum, but when Span 80 was used in concentrations exceeding 2.0% with the Carbowax, the ability to absorb water was greatly increased. Therefore, the optimum concentration of a Span must be specific for each mixture.

**p. 37, Jan. 1960.*

Antibacterial Synergism between the Halogenated Bis-Phenols and the Halogenated Aromatic Anilides and Carbanilides.

By D. R. Noel, R. E. Casel, W. M. Linfield, and L. A. Harriman.

Many antibacterial agents are being used in toilet soaps to degerm the human skin and thus inhibit the development of perspiration odor. For the most part, these chemicals have been members of a series of halogenated bisphenols of which hexachlorophene and bithionol are good examples. Other agents such as 3, 4, 4'-trichlorocarbanilide and 2-hydroxy-5-chlorobenzoic acid 3', 4'-dichloroanilide are also highly active in soaps.

Gould and Bosniak, using enzymes inhibition experments, concluded that an unhalogenated bisphenol in combination with hexachlorophene exerted a sparing action leaving the more highly active hexachlorophene to act more effectively on other cell enzymes. This effect is greater than the combined additive effect of the individual agents. Synergism has been found to exist with the following combinations: bithionol and tyrothricin, and dichlorophene and tyrothricin.

In the study found in the January, 1960 issue of *APPLIED MICROBIOLOGY*, Vol. 8 No. 1, the synergistic activity was tested on the chemicals per se by testing them in toilet soap, a nonionic syndet, and a non-soap anionic syndet. 3, 4, 4'-Trichlorocarbanilide and 3, 3', 4-trichlorocarbanilide both show synergistic action when combined with hexachlorophene (G-11-S), the sulfur analog of hexachlorophene (G-11-S), tetrachlorophene (G-5), bitionol (B), and 2, 2'-thiobis (4-chloro-6-methylphenol) (S-1). 2-Hydroxy-5-chlorobenzoic acid 3', 4'-dichloroanilide and 2-hydroxy-5-chlorobenzoic acid 4'-chloroanilide both cooperate synergistically with G-11, G-11-S, G-5, and B. No synergism was exhibited between 3, 4, 4'-trichlorocarbanilide or 3, 3' 4-trichlorocarbanilide with dichlorophene (G-4), nor 2-hydroxy-5-chlorobenzoic acid 3' 4'-dichloroanilide or 2-hydroxy-5-chlorobenzoic acid 4'-chloroanilide with G-4. These results indicate that synergism is a highly specific property. It was shown that this synergism was independent of the carrier used and that it was most active for the higher halogenated bisphenols with the trichlorocarbanilides and the chlorinated salicylanilides.

Comparative Toxicity Studies on Ten Antibiotics in Current Uses.

By A. L. Bacharach, Barbara J. Clark, Maran McCulloch and E. G. Tomich.

(*digested from Jour. of Pharmacy and Pharmacology Vol. XI No.* 12, *Dec.* 1959. *p.* 737).

The question arises, "How toxic are the different antibiotics currently in clinical use?" The answer to this question has been up to this time incomplete. Thus, the Glaxo Laboratories Ltd. decided to conduct experiments on the same strain of the same species of laboratory animal by four different routes using ten currently used antibiotics. Their aim was to compare their relative toxicities. The male and female A26 strain albino and the male and female fawn GFF strain mice were used. The LD50 values were calculated by the method of de Beer from the number of deaths occurring within seven days of treatment. The mice were administered graded doses by four different routes-intravenous, intraperitoneal, subcutaneous, and oral. All the solutions or suspensions of antibiotics were prepared immediately before use. The range of doses was chosen so as to lie on each side of the expected LD50. The preparations used were: Bacitracin, Chloramphenecol (Chloromycetin), Chlortetracycline hydrochloride (Aureomycin), Erthromycin (Erythrocin), Neomycin sulfate, Oxytetracycline hydrochloride (Terramycin), Sodium benzylpenicillin, Polymyxin B Sulfate (Aerosporin), Streptomycin sulphate, and Tetracycline hydrochloride (Tetracyn). Penicillin remains to be least toxic of all currently used antibiotics, by either the intraperitoneal or intravenous route. Streptomycin, neomycin, and penicillin have one-eighth to one-quarter the oral toxicity of the other seven antibiotics tested. Polymyxin appeared to be the most toxic by any route. Generally, the differences between the two strains was small. The subcutaneous toxicity of Tetracycline hydrochloride is decreased when it is administered as a neutralized suspension. Ata pH of 1.8, tetracycline hydrochloride produced severe necrosis at the site of injection, but at a neutral pH, it produces none.

Toxicity was shown to be less if given by the intraperitoneal route than by the intravenous route except for chlortetracycline and bacitracin. The intravenous injections of chloramphenecol, neomycin, and streptomycin were six or more times as toxic as the intraperitoneal route the corresponding ratios falling for erythromycin, polymyxin tetracycline, and chlortetracycline, in that order, to the reversed ratio for bacitracin. Only four of the antibiotics tested by the oral route showed the presence of strain differences. No marked differences were noted in strain-sex comparisons by any of the parenteral routes. They tended to be greatest by the sucutaneous and least by the intravenous route. By the subcutaneous route toxicities were either the same for both strain-sex groups or slightly to distinctly greater for A2G males. The evidence of the oral tests shows the difference to have been greater between the strains than the sexes for four of the antibiotics, namely, chloramphenecol, erythromycin, benzylpenicillin, and streptomycin. There is increasing evidence of a difference between strains of the same species as to the response of these animals to the same stimulus or stress. Consequently, it is necesary to give the fullest possible details about any animals used in research.

In an article in the March issue of American Perfumer titled "The scope of fatty acids and derivatives in cosmetic formulations", an error appeared in a formula on page 25, at the bottom of the right hand column. It should have appeared.

$$C_{13}H_{27}COO\text{-}CH_2\text{-}CH\text{-}C_6H_{13}$$
$$\qquad\qquad\qquad\qquad\quad |$$
$$\qquad\qquad\qquad\qquad C_8H_{17}$$

Toward the top of the same column Arachidonic acids was misspelled.

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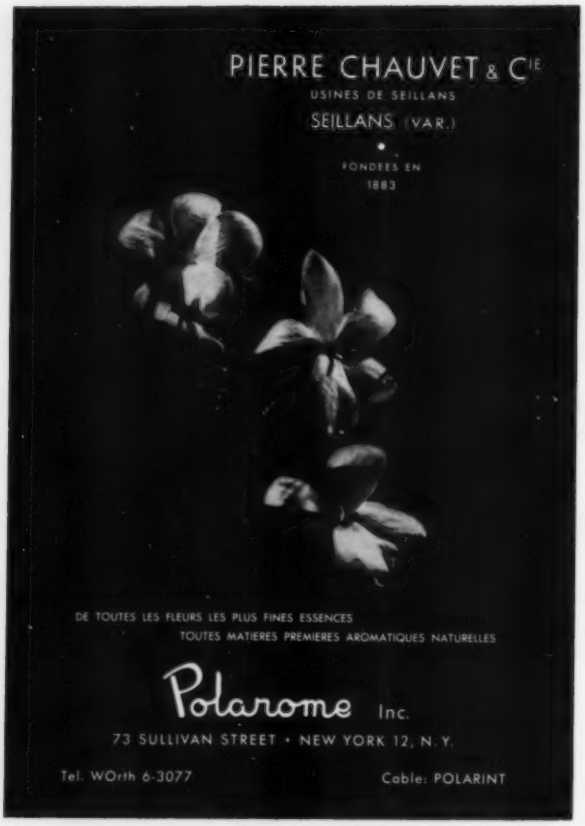

Dr. Erich Boehm dies

Co-inventor of the para hydroxy-benzoic acids

Dr. Erich Boehm, co-inventor of the para hydroxy-benzoic acids and gallate antioxidants, died suddenly at Cardiff on December 28, 1959. Dr. Boehm was educated in Berlin, beginning his studies in 1920 at the University of Berlin. In 1922 he graduated at Berlin and in 1925 he was awarded the degree of Doctor of Philosophy of the University of Berlin. His first post was that of a chemist with a food manufacturing firm in Berlin. It was about this time that Professor Sabalitschka, in collaboration with Heinrich Jacobson, one of the directors of this firm, patented the use of Esters of p-Hydroxy Benzoic Acid as preservatives, later to become known as the Nipa Esters.

It soon became apparent that the Nipa Esters were superior to all other substances hitherto proposed as non-toxic preservatives and their application was extended from pharmaceutical and medicinal products to Cosmetics and Foods and also to many technical fields.

Early in 1939, with others, Dr. Boehm formed the Company of Nipa Laboratories Ltd., in England.

The antioxidant activity of the esters of gallic acid had been discovered by Boehm and Sabalitschka in 1938, but only some members of the series had been prepared and then essentially by laboratory methods. One of the first tasks was to study the preparation of these compounds and to develop practical means of manufacturing them. At the same time, the earlier discoveries were checked and new applications investigated. The preliminary pharmacological tests were confirmed and extended by Drs. Coward and Dyer of the Pharmacological Laboratories of the Pharmaceutical Society of Great Britain. Their findings confirmed the non-toxicity of these compounds and indicated their suitability for use in pharmaceuticals, cosmetics and foods. The first paper published in Britian on these new and interesting substances which inhibit the development of rancidity in edible fats and oils, appeared in the Journal of Pharmacy and Pharmacology under the joint authorship of Boehm and Williams.

In 1942, Dr. Boehm discovered that certain mono-aryl and mono-aklyl ethers of ethylene glycol showed antibacterial activity.

Certain carefully formulated combinations of the Nipa Ester preservatives had been marketed for some years. But it is probably true to say that no-one appreciated the value and significance of these combinations as much as Dr. Boehm, and in more recent years he devoted much of his attention to the formulation of these mixtures which show an increasingly powerful and extended microbiological activity.

The whole of his working life was devoted to preservatives, antioxidants and antiseptics and his reputation and authority in this field received international recognition with his appointment to the Joint FAO/WHO Committee set up to consider the addition of chemical additives to Foods in 1958.

Dr. Boehm was not an ordinary man. He combined rare intellectual qualities with the integrity, charm and kindness of a true gentleman. He was always warmly interested in people as individuals regardless of their social standing. He will long be remembered by all who met him, but those who were privileged to know him well and to work closely with him will never forget him.

. . NEWS AND EVENTS . . . NEWS AND

Preliminary program of International Federation of SCC

The schedule for the Congress of the International Federation of Societies of Cosmetic Chemists to be held in Munich, August 31 to September 3, 1960, has been fairly well determined. The following papers have been registered for presentation.

Dr. H. Eder, Frankfurt: "Animal experiments concerning the effects of certain biological principles upon the release of energy." Dr. G. A. Erlemann, Basle: "Cosmetic additives (active principles)." Dr. H. Freytag, Darmstadt: "A contribution to the knowledge of physical and chemical behavior of human hair."

A. Herzka, B.Sc., London: "The aerosols". Dr. O. Jacobi, Wiesbaden: "Swelling of hair in cold-wave treatments—Investigations in polarized light." Dr. W. Keller, Berlin: "Hydroquinone monobenzyl ether as melanine inhibitor."

Prof. Dr. F. Neuwald, Hamburg: "IR spectrophotometric analysis of fats for cosmetic preparations." Dr. E. Paukner, Munich: "The effect of scents—olfactory psychology—A study of fundamentals." Dr. H. Schmitt, Cologne: Topic to be announced. Prof. L. Th. Schonagen, Leiden: "The application of cosmetic to preventive medicine."

Congress of Beauty Care and Cosmetology in the Netherlands

The fourteenth International Congress of Beauty Care and Cosmetology will be held in Amsterdam from the 5th to the 9th of September, 1960, under the auspices of the General Association of Beauty Parlours in the Netherlands. Experts from Holland and other countries will lecture on various topical problems concerning beauty care and cosmetology, and numerous demonstrations will enliven the Congress. An exhibition of beauty products and fittings for beauty parlours will be held.

Papers for SCC spring meeting

The spring meeting of the Society of Cosmetic Chemists will feature the presentation of provocative material discussing practical problems of the cosmetic chemist.

Keynoting the program will be Dr. G. Robert Clark, an honorary member of the Society, and director, Division of Cosmetics, Food and Drug Administration, Washington, D. C., who will outline current problems of concern to the administration and the industry.

Scientific papers will include the following: "The Pathogenesis of Alopecia", Dr. Norman Orentreich, New York City, "The Assay of Lyophilised Placenta Extract", Dr. William Colburn, Colburn Laboratories, Chicago, Illinois, "Effect of Vehicle Components and Sebum Constituents on the Absorption Characteristics of Sun Screens", Dr. Sydney Reigelman, College of Pharmacy, University of California, San Francisco, California, "Molecular Forces in Permanent Waving", Mr. Herman Bogaty, Associate research director, The Toni Company, Chicago, Illinois, "Non-Aqueous Foam Propulsion Systems", Mr. David S. Tillotson, Pennsalt Chemicals, Chestnut Hill, Pa., "The Use of Ternary Diagrams in Cosmetic Formulations", Mr. R. James and Mr. R. Goldenberg, Shulton, Inc., Clifton, N. J.

Union Carbide Chemicals to increase production of key chemicals

Union Carbide Chemicals has awarded a construction contract to alter and revise the plant facilities at Brownsville, Texas. Among the chemicals to be made at Brownsville are acetic acid, acetic anhydride, and methyl ethyl ketone. These chemicals will be made in large quantities to fill the demands of the current chemical market, particularly the rapidly growing surface coating, pharmaceutical and textile industries.

TGA Scientific Section meets May 11; to hear seven papers

The Scientific Section of the Toilet Goods Association will meet on Wednesday May 11th in the Sert Room of the Waldorf-Astoria Hotel, New York City and luncheon will be served in the Empire Room.

The following papers will be presented: "Program for Dermal Toxicity Testing" by Bernard Davidow, Ph.D., director of pharmacology, New Drug Institute, "The Rationale and Interpretation of Toxicity Tests", by Bernard Oser, Ph.D., president, Food and Drug Research Laboratories, Inc., "A Review of Product Development Problems under the Federal Food, Drug and Cosmetic Act" by Lloyd W. Hazelton, Ph.D., and John R. Elsea, Ph.D., Hazelton Laboratories, "Toxicological Aspects of Certain Types of Cosmetics" by A. J. Lehman, M.D., director, Division of Pharmacology, Bureau of Biological and Physical Sciences, Food and Drug Administration, Department of Health, Education and Welfare, "Usefulness of Rheology in Cosmetics" by Herbert Heinrich and John Clements, Coty, Inc., "Organoleptic Quality and the Stereochemical Theory of Olfaction" by James W. Johnston, Jr., Ph.D., Georgetown University, Schools of Medicine and Dentistry, Department of Physiology and Biophysics, "The Percutaneous Absorption of Estrogen" by Edward J. Masters, Ph.D., director of product research and development, Helena Rubinstein.

Atlas Powder names new board members

Atlas Powder Company has announced the election of Max E. Colson and Edward J. Massaglia to its board of directors. Mr. Colson is a vice president of Atlas and general manager of its Explosives Division. Mr. Massaglia is a vice president of Atlas and general manager of its Chemicals Division.

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Dr. Aaron Lerner

Aaron Lerner receives SCC Special Award

Dr. Aaron Lerner will receive the 1960 Special Award of the Society of Cosmetic Chemists and its prize of $1,000 in recognition of his work on enzymes and hormones controlling skin pigmentation. The presentation will be made on May 12, 1960 at the Hotel Biltmore in New York at the Society's semi-annual meeting.

Dr. Lerner, professor of the Yale University School of Medicine, is a pioneer in the study of melanin and was instrumental in the isolation of two melanocyte stimulating hormones from hog pituitaries. His work has demonstrated the similarity of these hormones obtained from man to that obtained from other species.

Noudex appoints Ritter and Seymour

Noudex Products Company, Division of Heyden Newport Chemical Corporation announced the appointment of Frank X. Ritter as regional sales manager, Central region, and William C. Seymour as regional sales manager, Eastern region. Both men served previously as regional sales representatives in their respective areas.

Fleuroma to modernize factory

Fleuroma, Inc. has announced the letting of a contract for the alterations of their new building and plant. Work has already begun and is expected to be completed by the middle of June.

American Plastic to add blow-molding

Blow-molding has been added to the line of custom-molding services offered by American Plastics Corporation, a subsidiary of Heyden Newport Chemical Corporation. The company will produce blow-molded bottles and shapes to custom requirements or supply a variety of oval-shaped squeeze bottles and applicator tips from stock molds.

Biddle Sawyer to represent Takasago Perfumery Co.

Biddle Sawyer Corporation has been appointed exclusive agents in the United States for Takasago Perfumery Co., Ltd., Tokyo, Japan, manufacturers of aromatic chemicals, essential oils and flavoring agents.

DeRagon joins American Cholesterol

Miss Shirley Ann DeRagon has been appointed formulation chemist in the technical service department of American Cholesterol Products, Inc. Miss DeRagon was formerly a research chemist at Johnson and Johnson with whom she was associated for the past ten years.

Snell acquires Sperling Laboratories

Foster D. Snell, Inc. has announced acquisition of Sperling Laboratories of Arlington, Virginia. Sperling has rendered services in the area of acute and chronic toxicity studies, preparation of petitions to FDA for clearance of drugs and food-additive chemicals, and a variety of similar services in the pharmacological field.

Parfums Marcel Rochas retains Jill Jessee

Parfums Marcel Rochas Inc. has appointed Jill Jessee, Inc. to handle the company's publicity. Jill Jessee whose firm specializes in cosmetic and perfume publicity, advertising and promotion represents a number of other companies in the industry.

Strong Cobb Arner Reports Sales Figures

Strong Cobb Arner, Inc., leading diversified customer-manufacturer of pharmaceutical and allied products, has reported to its stockholders that sales volume for the 10-months ended November 30, 1959 was almost 5% ahead of the corresponding periods of the predecessor companies in 1958. The improvement reflects a substantial increase in bookings and shipments since the merger of Strong, Cobb and Arner in mid-1958. SCA order backlogs are approximately 40% ahead of this time a year ago.

As a result of intensive engineering study the company has decided to continue operations in its present three domestic and two Canadian locations, and to expand and improve them to meet customer demands.

33% of Collapsible Metal Tubes Used for Cosmetics

Items packaged for the drug trade accounted for 85% of the collapsible metal tubes manufactured in 1959. Total sales of these fold-up tubes were up 14% from 1958. The largest gain (33%) was in fold-up metal tubes for cosmetics, with 1959 shipments totaling 104,187,024 compared with 78,069,024 in the preceding year.

This annual statistical report from The Collapsible Tube Manufacturers Council showed 973,197,504 tubes shipped for drug trade items compared to 838,004,256 in 1958. Toothpaste containers accounted for 50.5% of the total manufactured, maintaining their traditional lead in the use of collapsible metal tubes. Shipments of toothpaste tubes in 1959 increased 13% over the 1958 total.

Another major gain occurred in metal tubes for medical-pharmaceutical packaging. 1959 use increased 22% over 1958.

Shipments of shaving cream tubes declined 9% under 1958's total.

Strong Cobb Arner Begins Overseas Licensing Program

Strong Cobb Arner Inc. (formed by the merger of Arner Company and Strong, Cobb in 1958) has set up an overseas licensing program for manufacturing its matrix form of sustained release tablets. The first licensee, a Pfizer subsidiary, will be assisted in setting up manufacturing and quality control facilities by Dr. William E. Gaunt, vice president and scientific director of SCA, who is in Europe for that purpose and to discuss future license agreements with major drug firms in the European Common Market.

Obituary

Albin Wilko flavor chemist for Fries & Fries, Inc. of Cincinnati and New York, died on February 29th.

Mr. Wilko was a flavor chemist in the Eastern laboratory of the company. He was a veteran in the industry, having worked for more than a quarter century as a perfume and flavor chemist.

Mr. Hans-Erich Steche

Obituary

Mr. Hans-Erich Steche, director of Haarmann & Reimer GMBH, Germany has passed away.

Mr. Steche was born in Leipzig, Germany where his father was owner of Heine & Co. He was forced to take over the management of this company just after World War I as his father was compelled to retire because of ill health. He was successfully in charge of the company until the end of the second World War.

After the war only the plant at Leipzig, which was badly damaged by bombs, was left. Conditions of the following years, however, made the free management of the plant so difficult that the deceased moved to Western Germany with his family. He joined Haarmann & Reimer GMBH in the fall of 1949 and was appointed director of this company shortly afterwards.

In this capacity he worked successfully and energetically and employed his wide experiences and knowledge also in the Associations of the aromatic and flavor industry.

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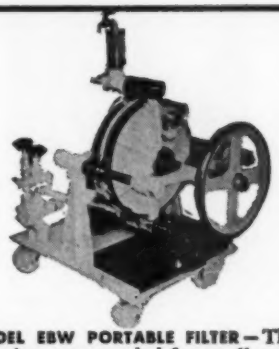

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PERSONALITIES

Mr. Harold N. Anderson

Harold N. Anderson has been promoted to manager of cosmetic color sales of H. Kohnstamm & Co., Inc. Mr. Anderson has been associated with the company since 1929.

Mr. William G. Foley

William G. Foley has been appointed vice president of Dragoco, Inc. Mr. Foley will have over-all responsibility for domestic sales of the firm.

Mr. Chester Firestein

Lee H. Bristol, chairman of the board of the Bristol-Myers Company, has been named chairman of the 1960 Special Gifts Campaign of the National Conference of Christians and Jews.

Mr. Carl-Wilhelm Gerberding

Carl-Wilhelm Berberding, founder and senior partner of the firm of Dragoco, Holzminden, was awarded the Cross of the Federal Order of Merit 1st Class for "specially meritorious services rendered to the people of the State." Mr. Gerberding is shown being invested with the decoration by the President of the Provincial Administration, Dr. Suermann.

Mrs. LaVange Toepfer has been appointed design assistant to Seymour Kent, art director of Dorothy Gray, Ltd. Mrs. Toepfer, who will work on Dorothy Gray and Ogilvie Sisters packaging, was formerly with Raymond Lowey, industrial design firm and, before that, with General Beauty Products.

Chester Firestein and Lee Rosene have been elected vice presidents of Max Factor & Co. Mr. Firestein, director of the firm's United States merchandising, is a member of the third general of the Max Factor family. He is a grandson of the late Max Factor, Sr., and the son of Max Firestein, executive vice president. Lee Rosene is director of United States sales for Max Factor & Co.

Dr. Christopher H. Costello

Dr. Christopher H. Costello has been appointed to the newly-created position of director of research for the Colgate Pharmaceutical Laboratories Division of the Colgate-Palmolive Company.

Dr. Costello will plan and direct all research and development work of the Pharmaceutical Division. He also will be responsible for the Division's scientific programs and technical personnel.

Prior to joining Colgate-Palmolive, Dr. Costello had been associated for the past ten years with the Columbus Pharmacal Company of Columbus, Ohio most recently as vice president and scientific director.

Harvey Kornhaber has been appointed chemist in the skin products research section of the toilet articles division's research and development department of Colgate-Palmolive Company.

Mr. Lee Rosene

Dr. John M. Longfellow

Dr. John M. Longfellow has been named director of research and development for The Mennen Company. He was formerly associated with Colgate-Palmolive, Inc. in their research and development division. Dr. Longfellow's primary responsibility will be the coordination of Mennen's overall research and development operation under the recently expanded marketing program. He will serve on Mennen's product development committee.

Frank W. Olson has been appointed a group leader in the toilet articles division's research and development department of the Colgate-Palmolive Company. Mr. Olson will head the products formulation group of the hair products research section.

Mr. Robert Elias

Robert Elias has been appointed sales manager for Felton Chemical Company. Mr. Elias, who has complete charge of all domestic sales activities, has been with the company for many years.

Bruce W. Johnson has been named assistant brand manager in the Lehn & Fink Products Corporation. Replacing him as assistant division general manager will be William S. Watchman, Jr. In his new position Mr. Johnson will assist Samuel F. Melcher, Jr., group brand manager, in supervising distribution, marketing, advertising and promotion for Lysol brand disinfectant, Hinds Honey and Almond Cream and new products developed within the division.

Mr. Bruce W. Johnson

Earl F. Copp, vice-president of The Risdon Manufacturing Co. for 25 years has retired from this position to become special consultant to the company. He will continue as a member of the firm's board of directors and executive committee. Mr. Copp directed the operations of Risdon's cosmetic, aerosol and metal goods divisions.

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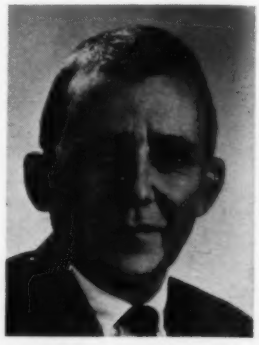
Mr. Marcel H. A. Billot

Marcel H. A. Billot recently retired from Houbigant, Paris. He was perfumer, technical and scientific general director of this firm with which he was associated for 35 years. Mr. Billot has announced that he does not intend to stop all activity but will be available as a consulting perfumer.

Dr. George R. Rowley has been appointed to the Colgate-Palmolive Company's Biological Research Laboratories. Dr. Rowley, who will work in the fields of biochemistry and enzymology joins Colgate from the College of Physicians and Surgeons of Columbia University where he served as an instructor of physiology.

Mr. Gustav Carsch

Gustav Carsch has been named technical service director of Roure Dupont, Inc. Mr. Carsch has previously been chief-perfumer of The Toni Co., chief-perfumer of Lanolin Plus, Inc. and branch manager of Polak & Schwarz Inc. in Chicago where he also served as chairman of the Society of Cosmetic Chemists.



Mr. Ames W. Howlett

Ames W. Howlett has been appointed assistant to the president of Bourjois, Inc. Mr. Howlett has represented Bourjois in the midwest since 1956 and prior to that was associated with Toni, Inc. and Hazel Bishop Inc. over a nine-year period of time.

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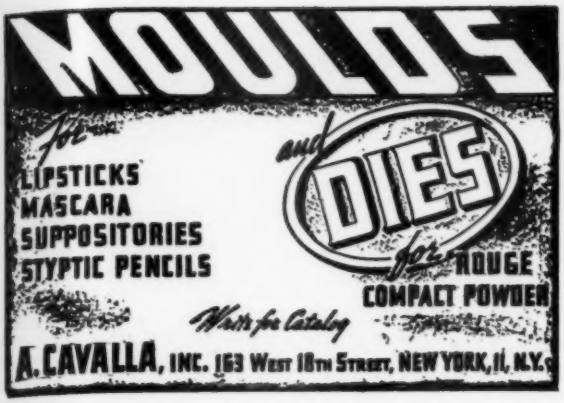

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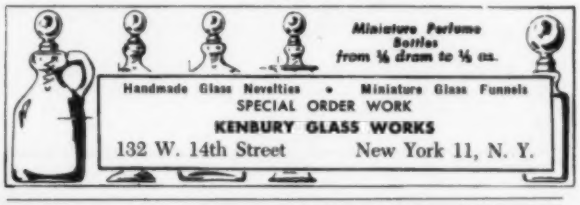
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